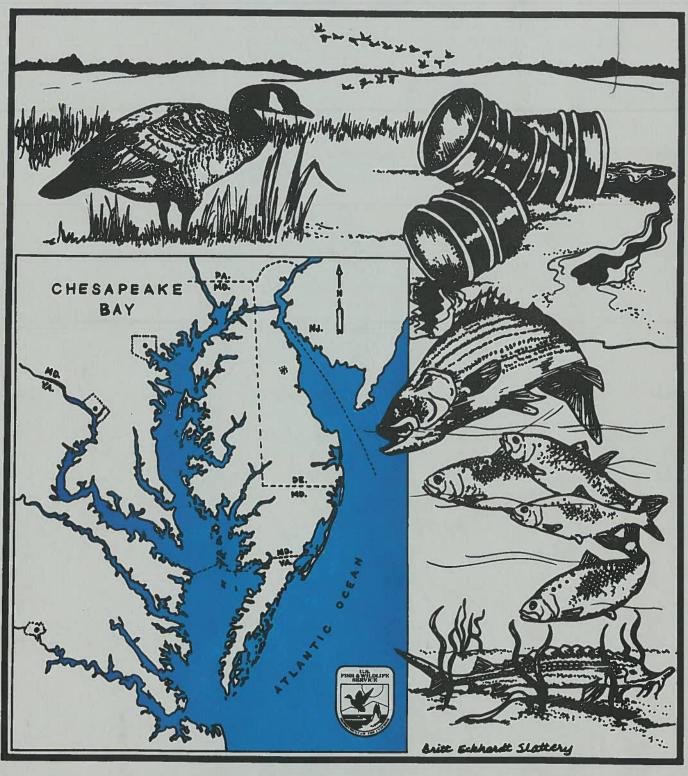
# A SURVEY OF CONTAMINANTS IN THE dup? **GREAT DISMAL SWAMP** NATIONAL WILDLIFE REFUGE, VIRGINIA



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A Survey of Contaminants in the Great Dismal Swamp National Wildlife Refuge, Virginia

U.S. Fish and Wildlife Service
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#### **ABSTRACT**

<u>Title</u>: A Survey of Contaminants in the Great Dismal Swamp National Wildlife Refuge, Virginia.

Abstract: A contaminant survey was conducted in the Great Dismal Swamp National Wildlife Refuge to determine the extent of contamination entering the Refuge from sources near its borders, including the Suffolk City Landfill Superfund site, several junkyards, and agricultural fields. The study analyzed metals and pesticides in surface water, and metals, pesticides, alkanes and polycyclic aromatic hydrcarbons (PAH) in small mammals, fish, sediment and soil.

The results from water, soil, and sediment samples indicate that the landfill and the junkyards may be sources of metal contamination to the Refuge. The results from small mammal samples indicate that the short-tailed shrew (Blarina brevicauda) and the white-footed mouse (Peromyscus leucopus), tend to bioaccumulate substances differentially, and this may be due to different food and living habits. The results of fish samples show that the species collected also tend to accumulate substances differentially and this is also probably due to food and living habits.

<u>Key words</u>: Environmental Contamination, Organochlorine Pesticides, Organophosphate Pesticides, Metals, Alkanes, PolycyclicAromatic Hydrocarbons (PAHs) Great Dismal Swamp National Wildlife Refuge, Dismal Swamp southeastern shrew (<u>Sorex longirostris fisheri</u>).

#### EXECUTIVE SUMMARY

The Great Dismal Swamp National Wildlife Refuge is located in southeastern Virginia and northeastern North Carolina and is bordered by a Superfund site, several automobile junkyards and agricultural fields. This study was conducted to determine if heavy metals and/or pesticides are entering the Refuge from these sources, and if so, to determine if they are being accumulated in the fish and small mammals located in the Refuge.

The study analyzed metals, pesticides, alkanes and polycyclic aromatic hydrocarbons (PAHs) in small mammals, fish, sediment and soil in 1987; and metals and pesticides in surface water in 1989; from sites selected for their proximity to the possible sources of contamination. From comparisons made between sites within the Refuge, the results indicate that the landfill and the junkyards may be sources of metals contamination to the Refuge.

The results also indicate that the short-tailed shrew (<u>Blarina brevicauda</u>) tends to have higher levels of metals than the white-footed mouse (<u>Peromyscus leucopus</u>), but that the mouse tends to have higher levels of alkanes than the shrew. These differences may be attributed to differences in these species' food sources. The results from fish samples show that a top predator, chain pickerel (<u>Esox niger</u>), had the highest concentrations of mercury and yellow bullheads (<u>Ictalurus natalis</u>), which live near the sediments, had the highest concentrations of chromium, nickel and iron. Golden shiner (<u>Notemigonus crysoleucas</u>) and yellow bullheads (<u>Ictalurus natalis</u>) were high in some alkanes.

This study provides data which can be used for future contaminant comparisons in the Refuge, and identifies the junkyards and landfill as probable sources of contaminant input to the Refuge. Future contaminant studies can be implemented in areas where there are high values to verify these values and to determine exactly what the sources of contamination are and how to reduce or eliminate them.

# INTRODUCTION

The Great Dismal Swamp National Wildlife Refuge (Refuge), created through the Dismal Swamp Act of 1974 (Public Law 93-402), is located in the Cities of Chesapeake and Suffolk in Virginia, and Camden, Pasquotank and Gates Counties in North Carolina. The Refuge is about 433 square kilometers or 43,300 hectares in size (Carter 1979), comprised mainly of forested wetlands with a 1,200 hectare shallow lake, Lake Drummond, in the central portion of the Refuge (Figure 1). Surface water in the swamp is acidic, ranging from pH 3.0 to 6.0. It has little buffering capacity, and is highly colored from humic substances and tannins.

The natural drainage patterns of the Refuge have been changed dramatically by a series of ditches which were cut in the 1700s and 1800s. These ditches were dug to facilitate logging of the forest, and in an attempt to drain the swamp to produce crops (Stewart 1979). In the late 1700s the larger Dismal Swamp Canal was begun on the eastern side of the swamp to facilitate commerce and transportation between Virginia and North Carolina. Due to financial constraints the canal was not made large enough for the volume of commerce until a major reconstruction took place in the early 1800s. Although commercial usage of the Dismal Swamp Canal later declined due to competition from the railroad, the canal is still in use today, mainly for recreational purposes (Stewart 1979).

The Refuge is bordered on the west and north by several automobile junkyards and agricultural fields. The Suffolk City Landfill site, which was placed on the National Priorities List under the Comprehensive Environmental Response Compensation and Liability Act in 1989 (making it a "Superfund site") is 1.5 miles upstream of the Refuge on the Pocosin Swamp which drains into Washington Ditch in the Refuge. The landfill received industrial and domestic waste, as well as 30 tons of organophosphate

pesticides which were buried on the site in the 1970s. All waters draining the landfill enter the Refuge. Automobile junkyards border the Refuge on the north, and are located adjacent to East Ditch which flows directly into the Refuge. Runoff from these junkyards due to spilled or leaking fluids or unclean fill may also impact the Refuge. The landfill, junk yards, and agricultural fields may all pose threats to the biota in the swamp through contaminated surface run-off.

This study was initiated to determine if heavy metals, alkanes, PAHs and/or pesticides are entering the Refuge from these sources, and if so, to determine if they are being accumulated in the fish and small mammals located in the swamp. Water and sediment samples from the ditches were also tested to determine which areas of the swamp are receiving input from these sources, what chemicals the input contains and at what concentrations the chemicals are present. This was intended to be a preliminary survey of contamination levels in the west and north sides of the Refuge.

The subspecies <u>Sorex longirostris fisheri</u>, the Dismal Swamp southeastern shrew, is listed as a threatened species by the U. S. Fish and Wildlife Service, and is endemic only to the historic swamp. Therefore, any impacts to small mammals as well as other mammals (Appendix A) in the swamp are of special interest. The bald eagle (<u>Haliaeetus leucocephalus</u>) is listed as an endangered species by the U. S. Fish and Wildlife Service and may eat fish and small mammals from the Refuge. Fish are also of special interest, since fishing is allowed on the lake, and people may consume their catch. See Appendix B for a list of fish species found on the Refuge. If fish are bioaccumulating contaminants these may be ingested by humans or by birds and mammals in the Refuge. The lake and ditches provide fishery resources for diving ducks, wading birds and recreational use.

#### MATERIALS AND METHODS

For the 1987 portion of the study, seven study sites were selected due to proximity to possible sources of contamination, including junkyards on the north of the Refuge, the Suffolk City Landfill Superfund site to the west, and agricultural fields along the west border (Figure 1). Badger Ditch was selected because it receives run-off from agricultural fields. Ditch was selected because it connects with Badger Ditch and flows toward Lake Drummond. The Cypress Swamp site was selected as a control site because it is not located near any apparent sources of contamination. East Ditch was selected because it is adjacent to the junkyards which may be a source of contamination. The Railroad Ditch site was selected because of its location in the central section of the swamp. Two sites were selected near the Suffolk Landfill site because of the possible contamination from the landfill. Washington Ditch was selected because it receives runoff from the landfill. Lake Drummond was selected because it receives runoff from many ditches and supports many species of fish.

Ten soil samples, eight sediment samples, fourteen fish samples and nine small mammal samples were collected for analysis in 1987 (Figure 2 and 3). Soil and sediment samples were collected near the mammal collection sites, and were shipped and analyzed along with the mammal samples. Both pitfall and live traps were used to trap the small mammals at each site. Pitfall traps were made of a #10 tin can, 6 inches by 9 inches, placed in holes in the ground so that the top of the can was flush with the surface of the ground. One to three inches of water were placed in each can so that small mammals that fell into the can could not jump out. Animals caught in live traps were killed either by cervical dislocation or thoracic compression. Animals were then placed separately into small labeled plastic bags, chilled in a cooler in the field and frozen after returning to the lab, until they

were examined for species identification, measurement, and skinning in preparation for the pollutant analysis. The whitefooted mouse (Peromyscus leucopus) and the short-tailed shrew (Blarina brevicauda) were chosen for tissue analysis due to their different food and living habits. Fish were caught using fish traps, fyke nets and gill nets. Fish samples were composited and homogenized according to the sample identification number on the sample label. Fish samples were then lyophilized, digested and analyzed for contaminants. Weyerhaeuser Analytical and Testing Services, Tacoma, Washington conducted organochlorine/arochlor and alkane analyses on these samples using gas chromatography. For complete lists of substances analyzed see Appendix C. Metals analyses on these samples were conducted by the University of Missouri Environmental Trace Substances Research Center, Columbia Missouri, using inductively coupled plasma (ICP) analysis, and mercury was confirmed using cold vapor atomic absorption. complete list of metals analyzed, see Appendix C. All samples were homogenized before analysis.

For the 1989 portion of the study, surface water samples were collected twice at 17 stations in Refuge ditches and 1 station in The first set was collected in June Lake Drummond (Figure 4). 1989, during high flow conditions, and the second set in July 1989, during low flow conditions. In the June sampling, the Cypress Swamp metal sample was broken during shipment and has not been included in the results. Three discrete, mid-depth water samples were collected at each sampling station (one 1-liter sample for organochlorine analysis, one 1-liter sample for organophosphate/carbamate analysis, and one 500 milliliter (ml) sample for metals analysis) in clean glass jars, using a water sampler which opened and closed at mid-depth. The sample for metals analysis was acidified in the field to pH  $\leq$  2 using reagent grade nitric acid. All samples for organic analyses were put on ice in the field and frozen at the end of the day.

Organophosphate/carbamate analyses on water samples were conducted using gas chromatography by the U.S. Fish and Wildlife Service's Patuxent Analytical Control Facility, Laurel, Maryland. For a complete list of substances analyzed see Appendix C. Organochlorine analyses were conducted using gas chromatography and fluorescence High Performance Liquid Chromatography (HPLC) by the Mississippi State Chemical Laboratory, Mississippi State, Mississippi. Metals analyses were conducted using ICP spectroscopy by Hazleton Laboratories America, Inc., Madison, Wisconsin.

#### RESULTS AND DISCUSSION

Because this study was conducted as a preliminary survey, small sample sizes were used to allow a maximum number of sites to be tested for a wide range of substances. Due to these small sample sizes, the samples have not been analyzed statistically. However, general areas of contamination within the Refuge can be seen.

Although not all metals, pesticides, alkanes and PAHs that were analyzed have associated quality criteria available, comparisons have been made with water and sediment quality standards that are available in the literature. This will help to assess the extent of contamination at sites that have elevated levels of certain substances when compared to the other samples in the Refuge. The study results can be used to formulate more detailed sampling plans for the Refuge.

# 1987 STUDY

## Small Mammals

In the small mammal samples, metals tended to be higher in Blarina than in Peromyscus when samples are compared within sample sites (Figure 5, Table 1). Blarina samples from East Ditch displayed elevated lead concentrations (47 ppm) compared to control values (18 ppm) reported in the same species by Beyer et The concentrations found at East Ditch were similar al. (1985). to those reported in house mice (Mus musculus, 42.3 ppm) from a lead contaminated site (O'Neil, 1988). In addition, mercury levels were higher in East Ditch shrews. Blarina had a concentration of 0.782 ppm, similar to levels found in Peromyscus spp. captured from fields where mercury-treated seed was used (approximately 0.808 ppm, Fimreite et al., 1970). Although these metal levels exceed normal ranges, many species do not exhibit any adverse effects in association with these elevated concentrations (Clark et al., 1992). Blarina trapped near East

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Ditch are high in several organochlorine pesticides (Figure 6, Table 2). Blarina may be higher than Peromyscus trapped at the same locations due to food habits, although it is not known what the source of the pesticides may be.

Alkane concentrations (Figure 7, Table 3) are generally higher in Peromyscus than Blarina when the two species are compared within This may be due to the different living and eating habits sites. of the two species. Because the endangered shrew, Sorex, has similar eating habits to Blarina it may not be impacted by alkanes as much as other small mammals with eating habits similar to <u>Peromyscus</u>. Alkanes are saturated aliphatic hydrocarbons and are constituents of petroleum products. They can come from highway runoff, automobile exhaust, and waste sites. Peromyscus may pick up more alkanes than Blarina if the alkanes are high in its food source, mainly vegetation; however the vegetation was not analyzed. Small mammal samples for PAH analyses were all near or below detection limits, therefore they are not shown graphically but are included in tabular form in Table 4.

<u>Peromyscus</u> spends a lot of time in trees and will voluntarily swim while <u>Blarina</u> spends most of its time on the ground and in shallow burrows. <u>Peromyscus</u> will eat insects but mainly relies on fruits, seeds, and leafy vegetation, while <u>Blarina</u> relies largely on insects and earthworms, snails, slugs, centipedes, millipedes, spiders, salamanders, snakes, birds, mice and other shrews (Schwartz and Schwartz, 1981). <u>Blarina</u> may show an increase in contaminant concentrations from bioaccumulation through the food chain due to its consumption of omnivores and herbivores that may have eaten contaminated foods.

<u>Sorex longirostris fisheri</u> is listed as a threatened species by the U. S. Fish and Wildlife Service and is endemic only to the historical reaches of the swamp, so any impacts to small mammals are of interest to protect this subspecies. This species eats

spiders, moth larvae, slugs and snails, centipedes, and vegetation; and lives in moist fields, brushy areas and woods. Thus, this species largely resembles <u>Blarina</u> in living and dietary habits, and it is assumed that it will be similarly impacted by contaminants in its habitat.

## Fish

In the fish samples, some metals were higher in some fish species (Figure 8, Table 5). All stations and species sampled, with the exception of fliers from East Ditch, displayed elevated mercury The national geometric mean mercury concentration was reported to be approximately 0.4 ppm (converted from wet weight assuming 75% moisture) (Schmitt and Brumbaugh, 1990). pickerel (Esox niger) had the highest concentrations of mercury, possibly due to the fact that chain pickerel are top predators and mercury is bioaccumulated through the food chain. concentrations (10 and 55 ppm) were considered high in Lake Drummond yellow bullhead (Ictalurus natalis) when these values exceeded concentrations found in fish from a contaminated site of the Savannah River (2.8-8.2 ppm converted from wet weight assuming 75% moisture) (Winger et al., 1990). Although there is very little water quality criteria for nickel, Jenkins (1980) reported nickel levels in fish from uncontaminated freshwater sites to be between <0.8-8.0 ppm (converted from wet weight assuming 75% moisture). Concentrations of nickel from yellow bullhead (26 ppm) from Lake Drummond were above this level. concentrations were also elevated in yellow bullhead from Lake Drummond (653 ppm), creek chubsucker from East Ditch (591 ppm), and golden shiners from Washington Ditch (488 ppm). concentrations were similar to iron levels found in fish from a contaminated site of the Savannah River (116-2868 ppm converted from wet weight assuming 75% moisture) (Winger et al., 1990). Phillips and Russo (1978) reported that iron residues do not cause toxic effects in many aquatic biota therefore these iron concentrations may be inconsequential. Yellow bullheads might be

high in chromium, nickel, and iron possibly due to the fact that these fish live in close association with the sediments and therefore may pick up these metals from the sediments. perch from Lake Drummond had selenium concentrations (3.5 ppm) slightly above the 85th percentile (3.3 ppm converted from wet weight assuming 75% moisture) obtained through the National Contaminant Biomonitoring Program (NCBP) (Schmitt and Brumbaugh, Zinc concentrations fell below the 85th percentile levels (185 ppm converted from wet weight assuming 75% moisture) reported by NCBP (Schmitt and Brumbaugh, 1990) with the exception of Lake Drummond chain pickerel (193 ppm). The variability of the concentrations of aluminum (ranging ND to 380 ppm) may be due to the presence of aluminum associated with sediment in the gut of the fish which was incorporated in the total analysis especially in benthic fish species (Brumbaugh and Kane, 1985). The golden shiner (Notemigonus crysoleucas) was high in some alkanes, and yellow bullheads (Ictalurus natalis) were high in n-Hexadecane (Figure 9, Table 6). The results of the PAH analysis show all compounds to be near or below detection limits, and are therefore not shown graphically, but are included in tabular form in Table 7. The results of the analyses of organochlorine compounds in fish (Table 8) showed organochlorine levels to be well below the geometric mean residue concentrations according the National Contaminant Biomonitoring Program (Schmitt et al., 1990).

## Soil

The soil samples near Washington Ditch and Cypress Swamp tended to be higher in metals than soil samples from other collection sites (Figure 10, Table 9). Washington Ditch receives run-off from the landfill and therefore the soil nearby may have enriched metal concentrations due to this run-off. The Cypress Swamp site was intended to be a control site because it is located upland of the landfill. Because this site is high in metals, it indicates that metal contamination in soils may be due to some

other source of contamination that has not yet been identified, but may be connected to run-off from nearby roads or agriculture. The results of the analyses of organochlorine compounds in soil samples are all near or below detection limits, and are therefore not shown graphically, but are included in tabular form in Table 10.

The results of alkane analyses in soils are given in Figure 11 and Table 11. Railroad Ditch has elevated levels of several alkanes in soil samples. This may come from the same unknown source that contaminated this area and probably Cypress Swamp with metals.

Polycyclic aromatic hydrocarbon (PAH) concentrations were below or near detection limits in the soil samples and are given in tabular form in Table 12.

#### Sediment

Sediment samples collected from East Ditch, Cypress Swamp, and Railroad Ditch tend to be higher in metals than other sampling sites (Figure 12, Table 13). East Ditch samples may be high in metals due to its proximity to several automobile junkyards on the north border of the Refuge. Sediment samples collected from East Ditch were above Wisconsin Department of Natural Resources suggested sediment quality criteria for arsenic (10  $\mu$ g/g), lead (50  $\mu$ g/g), and zinc (100  $\mu$ g/g), and above Ontario Ministry of Environment proposed sediment quality criteria for iron (2%) (Baudo et. al. 1990) and within the copper concentration (42 ppm) designated as moderately polluted (25-50 ppm) by the EPA in unpublished criteria for Great Lakes sediment (Beyer, 1990). There are car parts in this ditch that are rusting, and fluids from cars may have migrated into this ditch also. This may be a significant source of metal contamination to East Ditch The source of metals at the Cypress Swamp site is sediments. unknown. Railroad Ditch receives run-off from Cypress Swamp, and the unknown source of metal contamination to that area may also be affecting this ditch.

The results of the analyses of organochlorine compounds in sediment samples are all near or below detection limits, and are therefore not shown graphically, but are included in tabular form The results of alkane analyses for sediment samples in Table 14. are given in Figure 13 and Table 15. Sediment samples from Railroad Ditch and East Ditch had elevated levels of some The same source that contaminated Railroad Ditch soils probably contaminated the sediments. East Ditch sediments may be contaminated from the material in the ditch north of the Refuge border. PAH levels in sediment samples were below detection limits except the sample taken in East Ditch which was above detection limits for several PAHs (Table 16). Among these PAHs, phenanthrene concentrations found in East Ditch samples (540 ppb) were above the range of concentrations observed in industrial regions (17-252 ppb) (Long and Morgan, 1990). Fluoranthene levels found in East Ditch sediment (1850 ppb) were also above the 1000 ppb concentration reported to result in adverse effects in biological organisms (Long and Morgan, 1990). Pyrene levels were only slightly elevated. The remaining PAH samples were below detection limits.

#### 1989 SAMPLING

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## Surface Water

Water samples were taken in 1989 for the analysis of metals during the months of June (Figures 14, Figure 15 and Table 17) and July (Figures 16, Figure 17 and Table 18). Water samples taken in June indicate that Pocosin Swamp Sites 4 and 5 and Hall Pocosin Swamp are higher in most metals than the other sites. Water samples taken in July show the Pocosin Swamp Site 4 and the Hall Pocosin Swamp site to be high in metals in comparison to the

other sites sampled. Organophosphate, carbamate, and organochlorine analyses were all below detection limits in both the June and July, 1989 samples.

The waters of Hall Pocosin Swamp receive agricultural drainage from the surrounding area and this may have some influence on metal concentrations. The Pocosin Swamp sample sites receive surface run-off from the closed landfill, and metal concentrations are probably high due to this influence. differences between the June and July samples may be due to different flow regimes in the ditches, which may cause different flushing patterns from the surrounding area and different dilution processes. In comparing the levels found in the surface water to the U. S. Environmental Protection Agency's Water Quality Criteria, some metals were found to exceed the criteria. Lead criteria (3.2  $\mu$ g/l) was exceeded in June 1989 Hall Pocosin Copper criteria (12  $\mu$ g/l) was exceeded in June and July 1989 Hall Pocosin Swamp and July 1989 Pocosin Swamp Site 7 water samples (U.S. EPA 1985). July 1989 Railroad Ditch samples had elevated levels of selenium (50 ppb). This value was similar to levels reported by Eisler (1985) in sewage contaminated waters (50-280 ppb). Chromium concentrations at Hall Pocosin Swamp (21 ppb) and Pocosin Swamp 5 (40 ppb) were above the range of concentrations (1-10 ppb) listed for freshwater rivers and lakes (Eisler, 1986). However, these criteria are water hardness dependent and water hardness data is not available for these samples.

Although pesticides were known to have been dumped in the Suffolk City Landfill, they may not be leaching into the Pocosin Swamp and surrounding ditches. Since all water samples were below detection limits in organophosphate pesticides, the organophosphate pesticides may not be reaching the areas sampled or they had not recently been applied at the time of sampling. Organophosphate pesticides have a low environmental persistence

in comparison to organochlorine pesticides (Smith 1987).

#### CONCLUSIONS AND RECOMMENDATIONS

Blarina is generally higher than Peromyscus in metals at most sampling sites, and higher in organochlorine compounds near East Ditch. Peromyscus is generally higher than Blarina in alkanes. These differences between species may be due to different living and eating habits of the two species. In fish samples, chain pickerel were highest in mercury while yellow bullheads were highest in chromium, nickel, iron and n-hexadecane. Golden shiner was high in some alkanes. Chain pickerel may be high in mercury due to the fact that it is a top predator and mercury is bioaccumulated through the food chain. Bullheads may be higher in some metals and n-hexadecane due to its living near the sediments.

Soils near Washington Ditch and Cypress Swamp were higher in metals while Railroad Ditch was higher in most alkanes. Washington Ditch receives runoff from Suffolk City Landfill and this may be the source of metals in the soil. Sediments from East Ditch, Cypress Swamp, and Railroad Ditch were highest in metals and Railroad Ditch and East Ditch were highest in some alkanes. Contamination in East Ditch is probably due to the junkyards located near this ditch. The sources of contamination to Cypress Swamp and Railroad Ditch are unknown.

June 1989 water samples were highest in metals at Pocosin Swamp Sites 4 and 5 and Hall Pocosin Swamp sampling site. July 1989 samples were highest in metals in Pocosin Swamp Site 4 and Hall Pocosin Swamp. The source of metals to the Hall Pocosin Swamp sampling site is unknown, while the source of metals to the Pocosin Swamp sites are probably the Suffolk City Landfill. Differences in contaminant concentrations at different sampling times, such as Pocosin Swamp Site 5, may be due to different flow regimes and flushing patterns.

The data indicate that there may be contaminant input from the Suffolk City Landfill and this information should be used to help direct the remediation of this Superfund site to reduce such The high levels of metals in East Ditch inflows in the future. indicate that the junkyards are also probable sources of The worst of the contamination from the junkyards contamination. may have been eliminated however, through a voluntary cleanup of a drainage ditch which flows into East Ditch, by the owner of one of the junkyards. Samples of water and sediment were taken near this area of contamination in January 1992 to determine the severity of contamination and the extent to which it has migrated through the Refuge. Those results will be reported separately.

Although many sites and parameters have been measured, few conclusions can be drawn as to the severity of contamination and the biological implications resulting from the contamination. This is due to the fact that there is little available reference data especially for fish, small mammals, and soil for many of the compounds measured, and due to the fact that the small sample sizes and lack of replicates preclude statistical analysis and verification of extreme values. Sample sizes were small and samples were not replicated due to limited funding for the study. With further funding, areas of concern from this study can be resampled with replication to further assess contamination and biological implications.

Using mapped surface water flow directions in the Refuge (Figure 18), it is evident that run-off originating from the Suffolk City Landfill in the Pocosin Swamp area, which drains into Washington Ditch, can reach Lake Drummond. This is of concern because it would allow any contamination from the landfill to migrate across the Refuge, and may also impact the lake and aquatic resources. This map is also useful to suggest a long term monitoring plan for the Refuge. Because the water in the Refuge mainly flows from west to east, the western border should be a primary objective for surface water monitoring because inflows may contain contaminants from surrounding sources. The north border

should also be a primary objective for surface water monitoring. Although the water is mainly contained above the railroad berm, the water from East Ditch does flow south to that berm and then flows east and west into other parts of the Refuge. Therefore, any sources of contamination from the north border may also migrate through the north section of the refuge.

The data indicate areas of special concern which could be included in a long term monitoring plan for the Refuge. This long term plan should include annual or semi-annual contaminant monitoring along the north and west borders of the Refuge to screen for new sources of contamination and more frequent contaminant monitoring of the areas that have been found in this study to be sources. This will insure that the clean-up efforts are successful and that these previous sources do not continue to release contaminants. In the future, the proposed Biomonitoring of Environmental Status and Trends (BEST) program may be useful as a monitoring tool for the Refuge.

In order for a comprehensive, effective monitoring plan to be designed, consideration needs to be given to specific goals and objectives which are to be fulfilled by the plan. Questions that should be answered in the design process include:

- What is the main goal of implementing a long-term monitoring plan?
- What parameters would be most important in the monitoring?
- Is the intent of the plan to monitor new contaminant inputs to the Refuge or to monitor and establish a data base for the whole Refuge?
- What amount of staff and monetary resources will be available to implement the monitoring?
- What natural resources are to be protected or monitored by implementing the plan?

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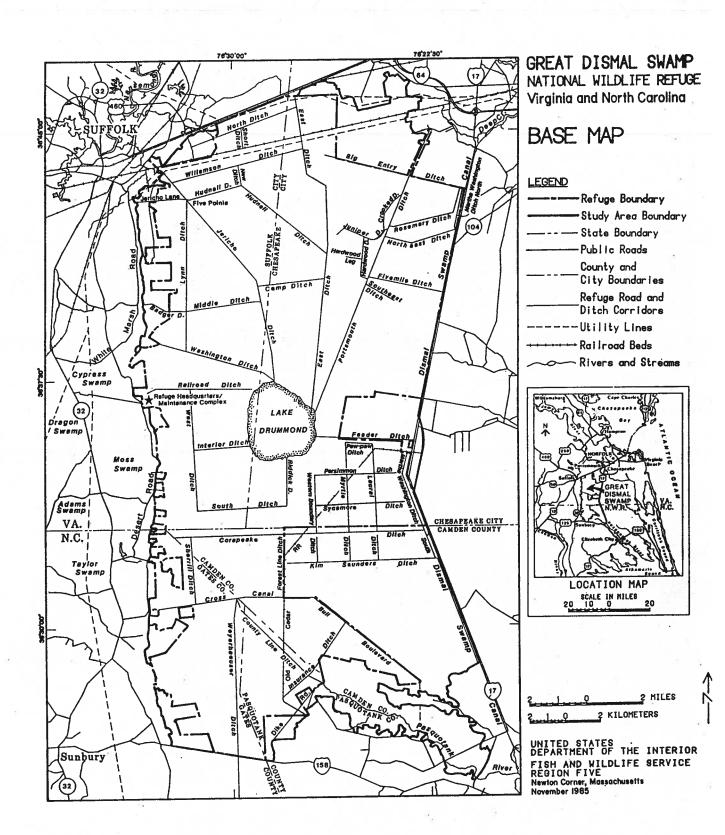


Figure 2: Small Mammal, Sediment and Soil Sampling Sites, July 1987.

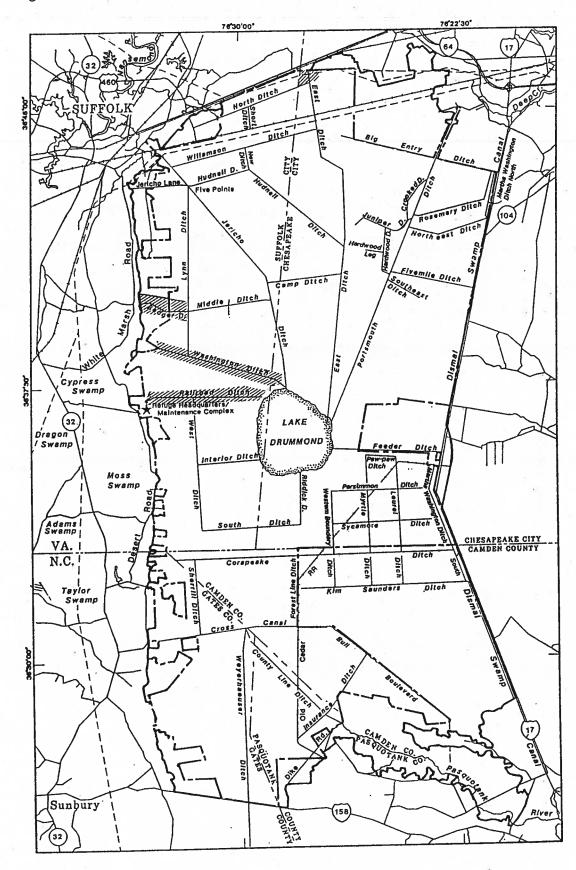


Figure 3: Fish Sampling Sites, July 1987.

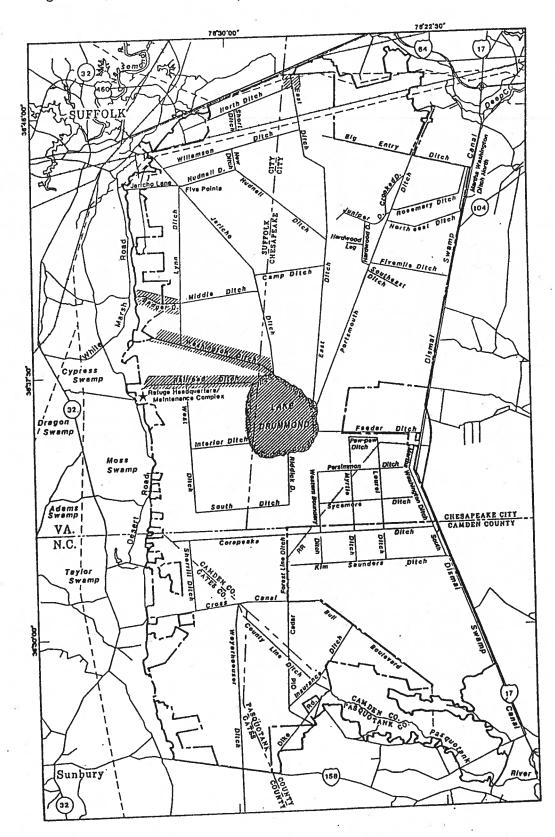
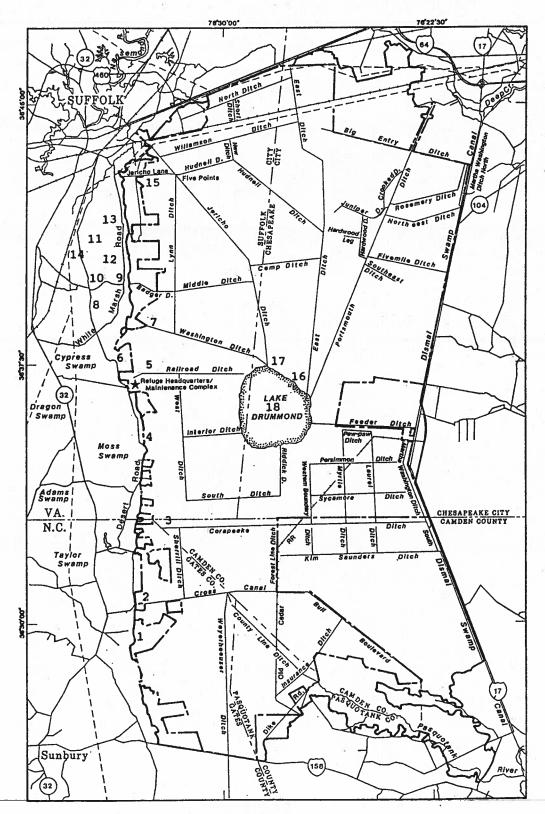


Figure 4: Water Sampling Sites, June and July 1989.

1= Hall Pocosin Swamp	7= Washington Ditch	13= Pocosin Swamp 6
2= Cross Canal	8= Pocosin Swamp 1	14= Pocosin Swamp 7
3= Corapeake Ditch	9= Pocosin Swamp 2	15= Jericho Ditch 1
4= Moss Swamp	10= Pocosin Swamp 3	16= East Ditch
5= Railroad Ditch	11= Pocosin Swamp 4	17= Jericho Ditch 2
6= Cypress Swamp	12= Pocosin Swamp 5	18= Lake Drummond



Ů> Figure 5. Metal concentrations in small mammals collected from Great Dismal Swamp National Wildlife Refuge, July 1987. PS1PM **PS1PM** Sample Site/Species Sample Site/Species RRPM RRPM WDBL Concentration ppm Concentration ppm EDPM 1,000 2,000 1,500 8 PM = Peromyscus leucopus WD = WASHINGTON DITCH BL = Blarina brevicauda CS = CYPRESS SWAMP PS = POCOSIN SWAMP RR = RAILROAD DITCH BD = BADGER DITCH ED = EAST DITCH Sample Sites Species Se **PS1BL** Sample Site/Species Sample Site/Species RRPM RRBL WDBL Concentration ppm Concentration ppm BDBL. EDBL 2 Ť 9

Organochlorine concentrations in small mammals collected from Great Dismal Swamp National Wildlife Refuge, July 1987. Figure 6.

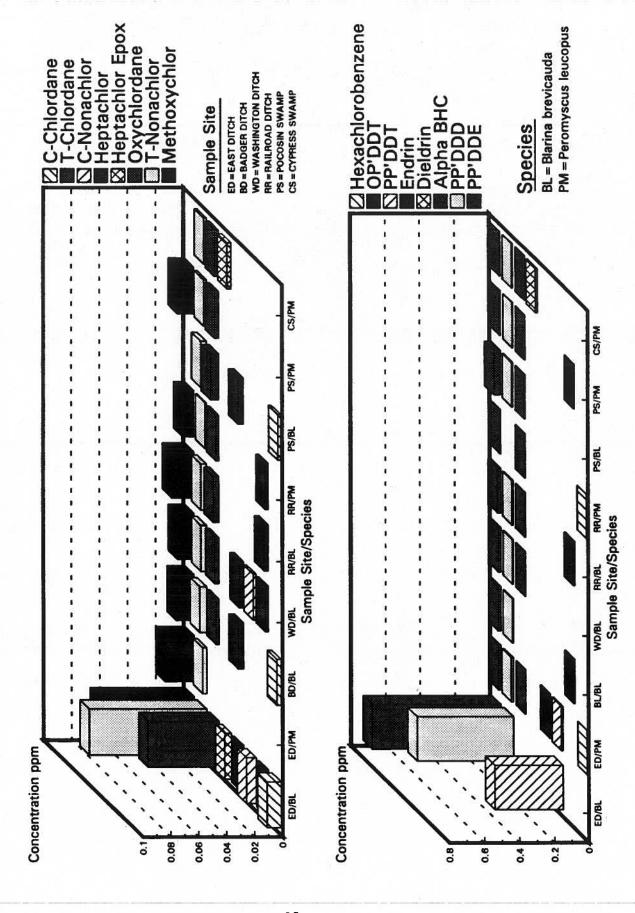


Figure 7. Alkane concentrations in small mammals collected from Great Dismal Swamp National Wildlife Refuge, July 1987.

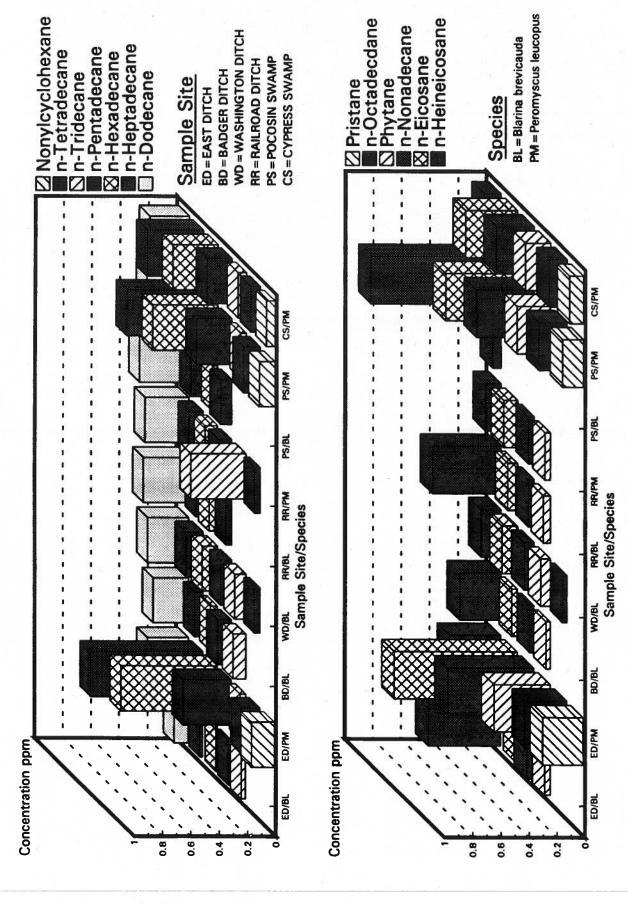
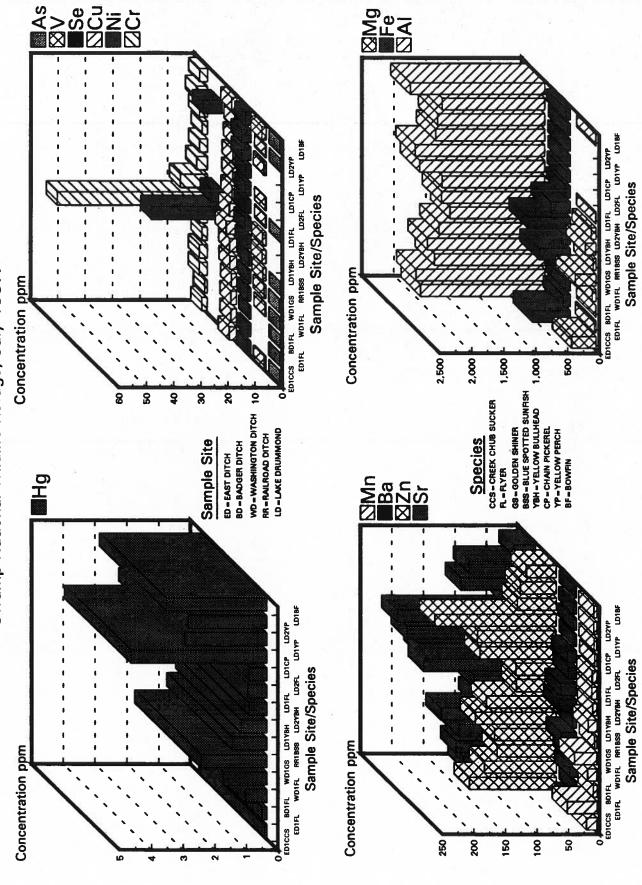
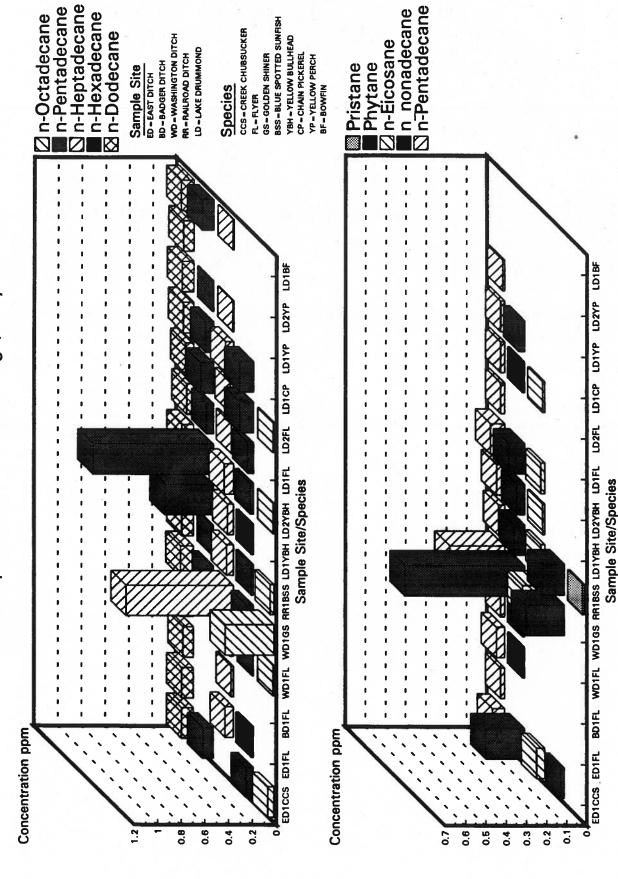


Figure 8. Metal concentrations in fish collected from Great Dismal Swamp National Wildlife Refuge, July 1987.

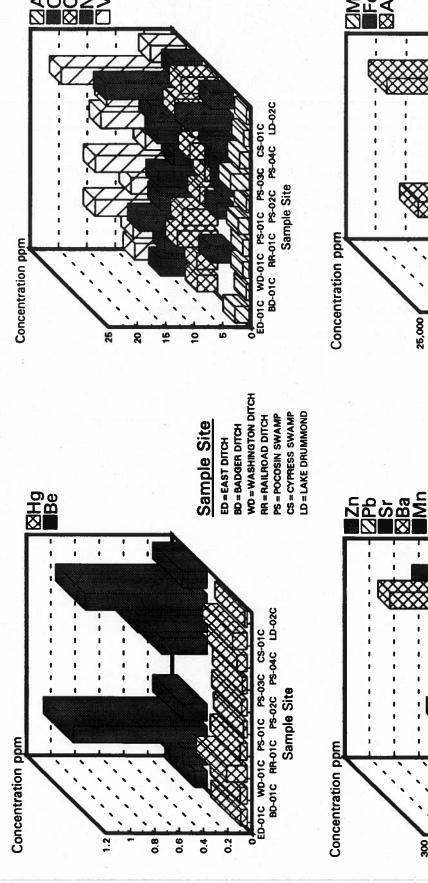


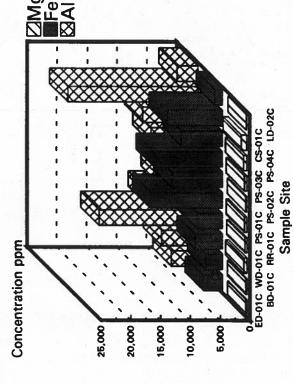
Alkane concentrations in fish collected from Great Dismal Swamp National Wildlife Refuge, July 1987. Figure 9.



37

Figure 10. Metal concentrations in soil collected from Great Dismal Swamp National Wildlife Refuge, August 1987.





ED-01C WD-01C PS-01C PS-03C CS-01C BD-01C RR-01C PS-02C PS-04C LD-02C

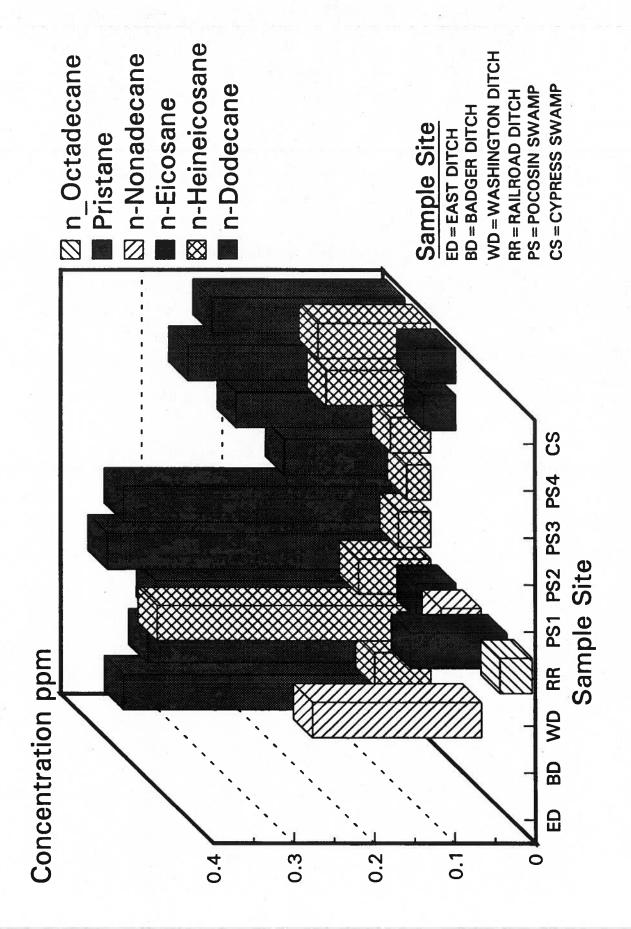
150

250

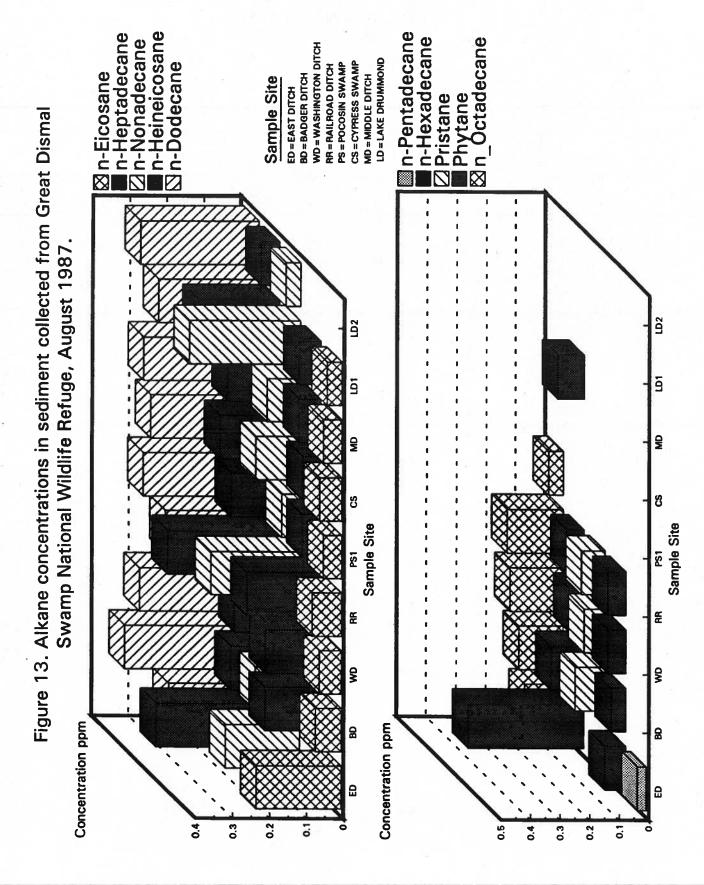
8 8

Sample Site

Figure 11. Alkane concentrations in soil collected from Great Dismal Swamp National Wildlife Refuge, August 1987



SCON SCON LD-01C Figure 12. Metal concentrations in sediment collected from Great Dismal Swamp National Wildlife Refuge, August 1987. CS-01C Sample Site Sample Site RR-01C Concentration ppm Concentration ppm BD-01C ED-01C 40,000 20,000 80,000 100,000 60,000 9 ജ 8 WD = WASHINGTON DITCH LD = LAKE DRUMMOND CS = CYPRESS SWAMP PS = POCOSIN SWAMP Sample Sites ED = EAST DITCH BD = BADGER DITCH MD = MIDDLE DITCH See See LD-01C LD-01C CS-01C RR-01C CS-0 Sample Site Sample Site PS-01C Concentration ppm Concentration ppm BD-01C 8 8 8 8 1.2 0.8 9.0 4.0



4/

Figure 14. Metal concentrations in water collected from the Great Dismal Swamp National Wildlife Refuge, June 1989

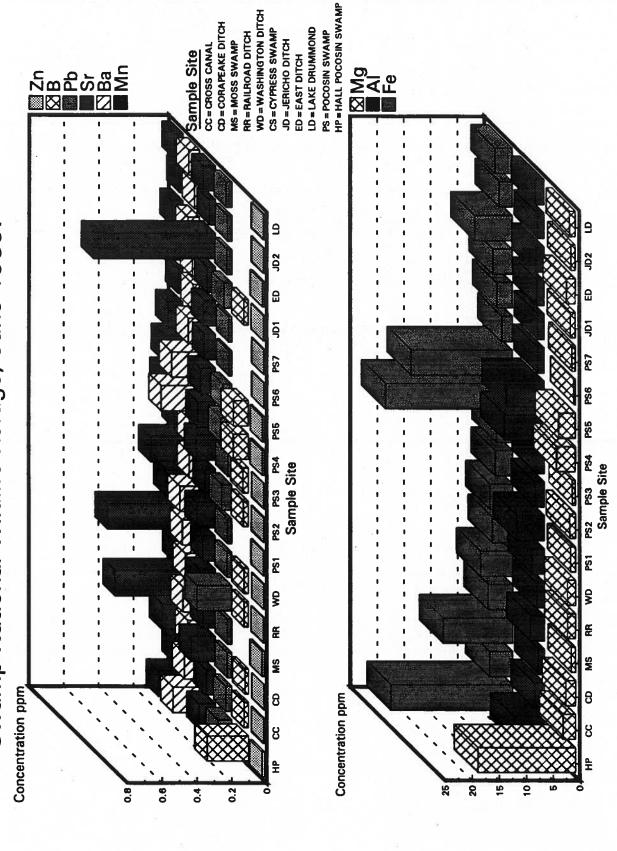
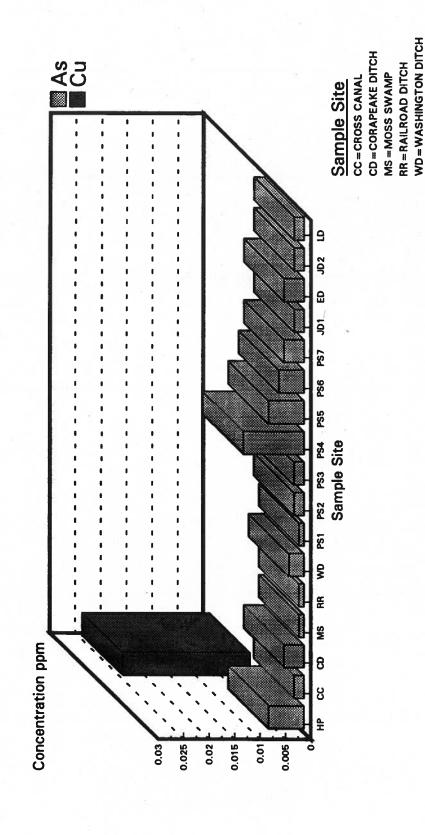


Figure 15. Metal concentrations in water collected from the Great Dismal Swamp National Wildlife Refuge, June 1989



PS = POCOSIN SWAMP
HP = HALL POCOSIN SWAMP

LD = LAKE DRUMMOND

CS = CYPRESS SWAMP

JD = JERICHO DITCH

ED = EAST DITCH

Figure 16. Metal concentrations in water collected from Great Dismal Swamp National Wildlife Refuge, July 1989.

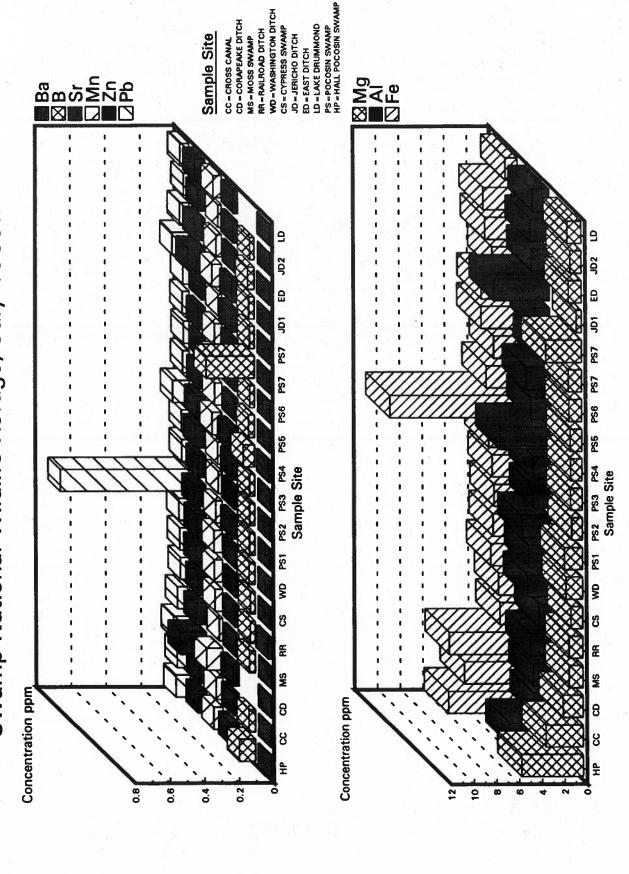
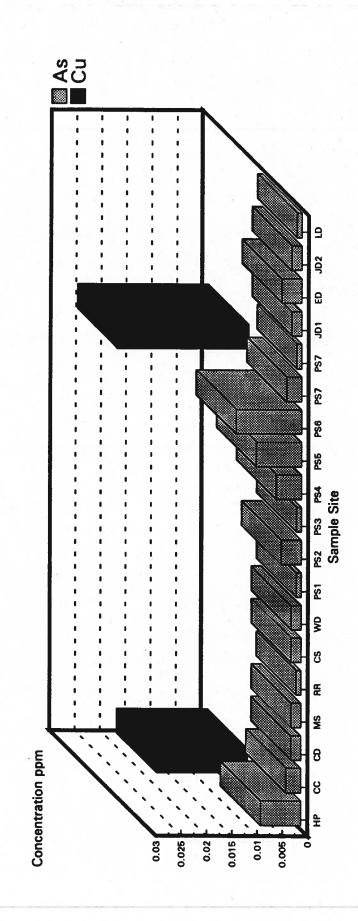


Figure 17. Metal concentrations in water collected from Great Dismal Swamp National Wildlife Refuge, July 1989.



# Sample Site

CC = CROSS CANAL
CD = CORAPEAKE DITCH
RR = RAILROAD DITCH
MS = MOSS SWAMP
WD = WASHINGTON DITCH
HP = HALL POCOSIN SWAMP

CS = CYPRESS SWAMP JD = JERICHO DITCH ED = EAST DITCH LD = LAKE DRUMMOND PS = POCOSIN SWAMP

Figure 18. Suggested sampling sites for long term contaminant monitoring

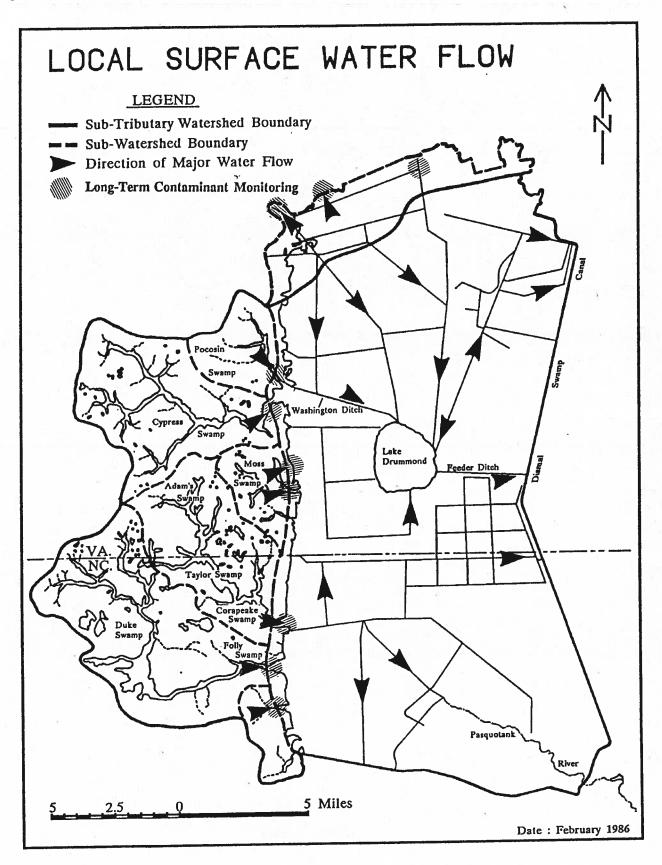


Table 1. Metal concentrations in small mammals from the Great Dismal Swamp National Wildlife Refuge, July 1987. Concentrations are in parts-per-million (dry-weight).

SITE	ED	ED	BD	WD	RR	RR	PS1	PS1	CS
SPECIES	BL	PM	BL	BL	BL	PM	BL	PM	PM
COMPOUN	<u>D</u>								
Se	1.8	1.1	1.6	1.4	2.6	0.78	1.6	0.9	0.99
As	ND	ND	0.64	ND	ND	ND	0.3	ND	ND
Hg	0.782	0.047	0.34	0.3	0.4	0.046	0.32	0.048	0.065
Ag	ND	ND	ND	ND	ND	ND	ND	ND	ND
Al	130	180	561	330	348	85	130	66	45
As	ND	ND	ND	ND	ND	ND	ND	ND	ND
В	ND	ND	ND	ND	ND	ND	ND	ND	ND
Ва	6.2	8.5	8.3	5.5	28.8	25.7	12.6	8.9	15.7
Ве	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cd	0.6	0.5	ND	ND	0.9	ND	ND	ND	ND
Cr	5.1	4.1	11	3	3.7	4.8	4.1	3	3
Cu	18	12	9.4	9.6	17	9.2	12	9.4	10
Fe	531	324	835	778	636	233	427	263	290
Mg	1290	1840	1090	1250	1330	1550	1330	1510	1750
Mn	6.5	16	10	8.8	9.7	16	20.2	8.6	11
Mo	ND	ND	ND	ND	ND	ND	ND	ND	ND
Ni	ND	ND	ND	ND	ND	ND	ND	ND	ND
Pb	47	8	ND	ND	8	ND	ND	5	ND
Se	ND	ND	ND	ND	ND	ND	ND	ND	ND
Sr	38.4	12.4	13.4	12.3	30.8	20.8	19.4	11	19
Tl	ND	ND	ND	ND	ND	ND	ND	ND	ND
v	0.5	0.3	0.7	0.6	ND	ND	ND ·	ND	ND
Zn	100	702	94.5	92.1	110	113	91.8	367	356

#### SPECIES

BL = Blarina brevicauda PM = Peromyscus leucopus

#### SITES

ED = EAST DITCH

BD = BADGER DITCH

WD = WASHINGTON DITCH

RR = RAILROAD DITCH

PS = POCOSIN SWAMP

CS = CYPRESS SWAMP

Organochlorine pesticide concentrations in small mammals from the Great Dismal Swamp National Wildlife Refuge, July 1987. Concentrations are in parts-per-million (wet-weight). Table 2.

ED = EAST DITCH BD = BADGER DITCH WD = WASHINGTON DITCH BL = Blarina brevicauda PM = Peromyscus leucopus SPECIES

SITES

CYPRESS SWAMP PS = POCOSIN -SWAMP CS = CYPRESS SWAMP

Alkane concentrations in small mammals from the Great Dismal Swamp National Wildlife Refuge, July, 1987. Concentrations are in parts-per-million (wet-weight). Table 3.

SITE	BL	ED PM	BL	WD	RR BL	RR PM	PS BL	S M	CS
COMPOUND									
n-Dodecane	0.1	0.29	0.25	0.29	0.32	0.31	0.35	0.3	0.28
n-Tridecane	0.03	0.04	0.09	0.08	ND	0.39	NO	0.04	90.0
n-Tetradecane	QN	0.13	ND	0.04	ND	0.04	ND	0.12	0.07
Octylcyclohexane	QN	QN	QN	QN	QN	QN	ON	QN	ND
n-Pentadecane	0.02	0.34	0.1	0.08	0.04	0.09	0.08	0.25	0.18
Nonvlcvclohexane	QN	0.16	ON	ND	NO	ND	QN	0.11	90.0
n-Hexadecane	0.01	0.68	0.05	0.11	90.0	0.09	0.04	0.46	0.32
n-Heptadecane	0.02	0.79	90.0	0.12	0.05	0.1	60.0	0.54	0.4
Pristane	QN	0.3	QN	0.03	ND	ND	QN	0.2	0.13
n-Octadecdane	0.01	0.87	0.04	0.11	0.07	0.1	QN	0.51	0.37
Phytane	QN	0.29	QN	ND	ND	QN	QN	0.15	0.1
n-Nonadecane	0.05	0.61	0.03	0.07	0.04	0.04	ND	0.41	0.27
n-Eicosane	0.04	0.4	0.03	0.07	0.05	0.04	ND	0.24	0.18
n-Heineicosane	0.51	0.36	0.29	0.14	0.48	0.11	90.0	0.92	0.12

PM = Peromyscus leucopus BL = Blarina brevicauda BD = BADGER DITCH WD = WASHINGTON DITCH RR = RAILROAD DITCH PS = POCOSIN SWAMP CS = CYPRESS SWAMP ED = EAST DITCH SITES

SPECIES

Dismal Swamp National weight). Table 4.

5	Polycyclic aromatic hydrocarbon concentrations Wildlife Refuge, July 1987. Concentrations are	drocarbon 1987. Co	conc	on concentratic Concentrations	tions ns a	ဌ구	small n n parts-	mammals s-per-mi	from	Great I	i t
	SITE	ED	ED	BD	WD	RR	RR	PS	PS	cs	
	SPECIES	BĽ	PM	BĽ	BL	BI	PM	BĽ	PM	PM	
	COMPOUND										
	Naphthalene	ND	ND	ND	Q	ND	ND	Q	Q.	ND	
	Acenaphthylene	ND	ND	NO	R	ND	ND	ND	QN	NO	
	Acenaphthene	ND	ND	NO	R	ND	ND	Q	QN	NO	
	Fluorene	ND	ND	N	ND	ND	ND	NO	NO	QN	
	Phenanthrene	0.02	ND	ND	Q.	90.0	N Q	Ω	ND	QN	
	Anthracene	ND	ND	Q	Q Q	N N	ND	N	NO	ND	
	Fluoranthene	ND	QN	QN	ND	QN	NO	QN	QN	ND	
	Pyrene	QN	ND	ND	R	ND	ND	ND	QN	NO	
	Benzo(a)anthracene	ND	QN	ND	Q.	ND	N	ND	ND	ND	
	Chrysene	ND	ND	ND	N	QN	ND	QN	ND	ND	
	Benzo(b)fluoranthene	ND	ND	ND	Q	ND	ND	QN	ND	NO	
	Benzo(k)fluoranthene	ND	QN	ND	S	QN	ND	QN	ND	NO	
	Benzo(a)pyrene	ND	NO	ND	Q	ND	ND	N	NO	NO	
	Benzo(e)pyrene	ND	ND	ND	ND	ND	ND	Q.	NO	QN	
	Perylene	NO	ND	ND	ND	ND	N	ND	QN	NO	
	Indeno(1,2,3-cd)pyrene	e ND	ND	ND	Q	ND	QN	QN	ND	ND	
	Dibenz(ah)anthracene	QN	NO	NO	ND	QN	Q	QN	ND	N	
	Benzo(ghi)perylene	QN	N Q	Q	ND	N Q	Q	N	ND	S	

SITES

ED = EAST DITCH
BD = BADGER DITCH
WD = WASHINGTON DITCH
RR = RAILROAD DITCH
PS = POCOSIN SWAMP
CS = CYPRESS SWAMP

BL = Blarina brevicauda PM = Peromyscus leucopus

SPECIES

1987.	
July	
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t Dismal	(dry-weight)
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concent	tration
Metal	Concen
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Table	

	3	concentrations are in parts-per-million	1S are in	parts-p	er-mililion	(ary-weignt)	igne).							
SITE	ED	ED	BD	QM	M.	RR	CI.	ΓD	9	LD CJ	E3	CI	G	CI
SPECIES	s ccs	F	H	FL	GS	BSS	YBH1	YBH2	FL1	FL2	CB	YP1	XP2	BF
Compound	nd													
Se	1.3	1.3	0.64	1.2	0.95	0.91	1.9	2.4	2.4	2.2	2.0	3.5	3.5	2.1
As	0.2	0.2	0.1	0.2	0.33	0.1	QN	QN	0.1	ND	QN	ND	0.1	0.2
Hg	0.083	0.170	0.675	1.4	2.1	1.1	0.815	0.573	0.604	0.567	4.3	2.6	2.5	3.2
Ag	ON	QN	ON ·	ND	ND	ND	NO	QN	ND	ND	QN	ND	ON	QN
Al	386	28	ω	62	332	49	170	78	S	ND	QN	ND	QN	ഹ
As	QN	QN	ND	ND	ND	ND	ND	NO	ND	QN	QN	QN	QN	QN
ш	ND	QN	ND	QN	ND	5.0	QN	6.5	6.3	5.0	ND	NO	QN	ND
Ва	10.5	3.8	11.2	13.3	24.3	6.4	27.8	26.0	13.3	15.3	3.5	5.7	4.4	3.1
Be	ND	QN	QN	ND	QN	QN	QN	QN	ND	QN	ND	ND	ND	ND
ੲ	QN	ND	ND	ND	ND	ND	QN	QN	ND	QN	ND	ND	ND	ND
Cr	2.0	1.0	3.0	3.0	2.0	3.0	55.0	10.0	4.0	3.0	2.0	3.0	4.2	3.0
ວ	2.7	2.1	2.1	1.9	2.5	2.6	3.9	2.7	1.8	1.5	1.4	0.89	0.72	1.9
Œ4 O)	591	123	83	06	488	140	653	309	73	64	48	51	42	74
Mg	1940	2010	1720	1830	1650	1480	1610	1600	1950	2020	1700	1670	1600	2100
Mn	17	48	9	15	36	4	14	9	ω	7	m	ß	2	S
Mo	ON	ND	ND CN	ND	ND	UND	QN	ΩN	ON	QN	NO	ND	ND	QN
N.	ND	ON	QN	ON	QN	ON	26	4	QN	ND	NO	ND	7	QN
Pb	QN	QN	QN	ND	ND	QN	QN	QN	QN	ND	QN	ND	ND	ON
Se	QN QN	QN	QN	QN	ND	ΩN	QN	ΩN	ND	ND	QN Q	5.0	ND	QN
Sr	115.0	127.0	123.0	150.0	98.2	97.8	178.0	185.0	205.0	220.0	46.6	119.0	106.0	34.0
ij	NO	QN	QN	ND	ND	ON	ND	ΩN	QN	QN	ND	QN	ND	QN
>	0.6	QN	QN	0.4	0.7	1.2	1.5	1.3	0.3	0.3	QN	0.8	0.3	1.6
Zn	138.0	114.0	93.6	97.9	128.0	111.0	62.9	63.6	121.0	125.0	193.0	61.7	56.9	47.4
									SP	SPECIES				
NO III	below	below detection limit	limit						CCS = CR	CREEK CHUBSUCKER	JCKER			
				SITES	ES				FL = FLIER	ER				
				ED = EA	EAST DITCH				GS = GOL	GOLDEN SHINER	~			
				BD = BA	BADGER DITCE	ir:			BSS = BL	BLUE SPOTTED	SUNFISH			
				WD = WA	WASHINGTON D	DITCH			YBH = YE	YELLOW BULLHEAD	TEAD			
				RR = RA	RAILROAD DITCH	тсн			CP = CHA	CHAIN PICKEREL	12			
				LD = LA	LAKE DRUMMON	Q.			YP = YEL	YELLOW PERCH				
									BF = BOWFIN	FIN				

Alkane concentrations in fish from the Great Dismal Swamp National Wildlife Refuge, July 1987. Concentrations are in parts-per-million (wet-weight). Table 6.

LD BF		0.11	Q	QN	Q	Q	QN	0.1	0.01	Q	Q	Q	S	Q.	N
LD YP2		0.09	ND	QN	QN	Q	QN	N	QN	Q	N	N	ND	N	N Q
LD YP1		0.1	ND	ND	QN	QN	QN	0.01	0.01	Q	QN	QN	QN	Q	0.01
G 6		0.09	QN	QN	QN	0.12	QN	0.07	0.07	Q	Q	N	0.03	S	0.02
LD FL2		0.07	QN	ND	ND	0.12	ND	0.11	0.03	N	0.01	NO	0.01	0.01	0.02
LD FL1		90.0	ND	ND	ND	0.05	QN	90.0	0.02	ND	ND	QN	N	ON	0.02
LD YBH2		0.1	Q	QN	QN	0.03	QN	1.02	0.08	QN	0.01	ND	0.08	0.02	0.07
LD YBH1		0.08	ND	ND	ND	0.02	Ω	0.41	0.05	ND	Q	ΩN	0.04	0.01	0.04
RR		0.1	ND	QN	N	0.02	N	0.01	90.0	0.01	0.03	0.11	0.05	0.02	0.03
WD		0.11	QN	ΝΩ	ΝΩ	90.0	ΩN	0.05	0.91	NO	0.41	0.19	0.59	0.11	0.27
ğ Ţ		0.11	QN	ΩN	QN	0.01	QN	QN	0.03	Q	0.01	N	0.01	QN	0.02
ET.		0.09	ND	ND	ND	Q	QN	QN	0.02	N	QN	NO	0.01	QN	0.04
8 F		0.1	QN	Q.	8	0.02	Q	S	0.07	Q	£	S	Q	S	0.02
ED		0.1	QN	S	QN (S)	90.0	ND	0.09	6.97	8	0.05	0.02	0.19	0.04	90.0
SITE	COMPOUND	n-Dodecane	n-Tridecane	n-Tetradecane	Octylcyclohexane	n-Pentadecane	Nonylcyclohexane	n-Hexadecane	n-Heptadecane	Pristane	n-Octadecane	Phytane	n_nonadecane	n-Eicosane	n-Heineicosane

ED = EAST DITCH

BD = BADGER DITCH

WD = WASHINGTON DITCH

RR = RAILROAD DITCH

LD = LAKE DRUMMOND

SET = COLDEN SHINER

BSS = BLUE SPOTTED SUNFISH

RP = YELLOW PERCH

BF = BOWFIN

Polycyclic aromatic hydrocarbon concentrations in fish from the Great Dismal Swamp National Wildlife Refuge, July 1987. Concentrations are in parts-per-million (wet-weight). Table 7.

				•	•					1	4		II s		
	SITE	ED	ED	ВО	WD	WD	RR	LD1	LD2	LD1	LD2	I.D	LD1	LD2	13
	SPECIES	SOO	Ę	Ę	Ę	GS	BSS	YBH	хвн	FL	F	GP CP	YP	YP	BF
	COMPOUND														
	Naphthalene 0	0.03	ND	QN	ND	0.02	QN	ND	NO	ND	0.02	0.02	QN	ND	Q
	Acenaphthylene	ND	NO	ND	NO	NO	Q	ND	ND	ND	ND	NO	ND	ND	Q
	Acenaphthene	ND	ND	ND	ND	QN	QN	ND	QN	NO	ND	N	ND	QN	N
	Fluorene	ND	N	ND	ND	ND	Q	ND	QN	ON	ON	N	ON	N	QN
	Phenanthrene 0	0.02 0	0.02	0.03	0.02	NO	NO	ND	QN	ND	NO	NO	NO	ND	QN
	Anthracene	ND	N	N	NO	N	ND	ND	ND	QN	ND	Q	N	QN	N
_2	Fluoranthene	ND	ON .	ΩN	QN	ND	N	ND	N	Q	ND	QN	N	N	N
	Pyrene	ND	ND	ND	N	ND	ND	NO	ND	ND	ND	N	NO	QN	Q
	Benzo(a)anthracene	ND	ND	ND	QN	ND	ND	ND	R	ND	ND	NO	ND	ND	QN
	Chrysene	ND	ND	ND	Q	QN	ND	ND	ND	ΩN	QN	ND	ND	N	Q
	Benzo(b)fluoranthene	ND	Q	QN	N	ND	ND	ND	QN	ND	ND	Q.	ND	QN	Q
	Benzo(k)fluoranthene	ND	QN	ND	QN	ND	NO	NO	QN	ND	ND	QN QN	ND	QN	Q
	Benzo(a)pyrene	NO	ΩN	N	N Q	ND	ND	ND	ND	ND	ND	QN	NO	ON	Q
	Benzo(e)pyrene	QN	N	Q	Q	ND	ND	ND	ND	ND	ND	Q	NO	UN	N
	Perylene	ND	NO	Q	ND	Q	ND	ND	ND	ND	ND	NO	NO	QN	S
	Indeno(1,2,3-cd)pyrene	QN	NO	ND	ND	NO	NO	NO	N	N Q	QN	Q	N	ND	R
	Dibenz(ah)anthracene	ΩN	NO	ND	QN	ND	ND	Q	ND	ND	ND	NO	ND	QN	ND
	Benzo(ghi)perylene	ND	ND	ND	ND	ND I	ND	NO	N	ND	QN	QN	ND	ND	ND

CS = CREEK CHUBSUCKER

'L = FLIER

GS = GOLDEN SHINER

BSS = BLUE SPOTTED SUNFISH YBH = YELLOW BULLHEAD

CP = YELLOW BULLHEAI CP = CHAIN PICKEREL

YP = YELLOW PERCH BF = BOWFIN

ED = EAST DITCH
BD = BADGER DITCH
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WD = WASHINGTON DITCH
LD = LAKE DRUMMOND

Organochlorine concentrations in fish from the Great Dismal Swamp National Wildlife Refuge, July 1987. Concentrations are in parts-per-million (wet-weight). Table 8.

LD		0.004	0.002	0.002	0.002	0.007	0.002	ND	0.008	0.042	0.027	0.005	ND	0.002	0.001	ND	0.044									
LD YP2		0.003	0.005	0.003	0.004	0.01	0.003	QN	0.011	0.045	0.048	0.008	QN	0.003	0.001	QN	0.075						FISH			
LD YP1		0.002	0.003	0.002	0.003	0.008	0.002	QN	0.009	0.038	0.039	0.007	QN	0.002	0.001	QN	0.049		CHICAGO	CREEK CHIRSHCKER		HINER	BLUE SPOTTED SUNFISH	YELLOW BULLHEAD	CKEREL	ERCH
g g		0.003	0.002	0.002	0.002	0.003	0.002	ON	0.007	0.013	0.009	0.005	QN	0.003	0.001	ON	0.026		S. C.	CREEK C	بتإ		= BLUE SP	= YELLOW		SOWFIN
LD FL2		0.002	0.002	0.002	0.002	0.003	0.003	QN	ND	0.014	0.012	0.004	QN	0.003	0.001	ND	0.026			800		GS =	BSS	171		A EE
ET.1		0.002	0.002	0.002	0.001	0.003	0.003	N N	0.01	0.011	0.01	ND	QN	0.002	0.001	Q	0.077									
LD YBH2		0.002	0.002	0.003	ND	0.002	0.002	8	0.007	0.008	0.007	0.007	QN	ND	Q	0.002	ND					гсн	bei			
LD YBH1		0.003	0.002	0.002	QN	0.003	0.003	S S	0.008	0.011	0.008	0.003	QN	ND	ND	ND	ND			HO#1	DITCH	WASHINGTON DITCH	RAILROAD DITCH	LAKE DRUMMOND		
RR		0.002	0.002	0.002	0.001	0.002	0.003	Q	ND	0.005	0.003	0.003	Q	0.003	0.001	QN -	ΝΩ		ST-T-S	EACT DITCH	BADGER DITCH	WASHIN	RAILRO	LAKE D		
WD GS		0.004	0.003	0.004	0.002	0.004	0.004	Q	Q	0.013	900.0	0.005	Q	QN Q	0.002	ND	QN			E C		WD ==	RR =	ii Ci		
F 7		0.002	0.002	0.002	0.001	0.002	0.003	QN	QN	0.01	0.004	900.0	ND	0.002	0.001	ND ND	QN Q									
80 71		0.002	0.002	0.002	0.002	0.003	0.003	QN	ON	0.01	0.004	0.008	QN	0.003	QN	QN	ND									
7 E		0.003	0.005	0.004	0.003	900.0	QN	0.002	0.008	0.009	0.009	Q	0.012	Q.	ND	QN	Q	limits								
ED CCS		0.002	0.003	0.003	0.002	0.003	0.003	0.002	Q	0.005	0.006	0.004	0.007	0.002	QN	QN	Q	ection								
SITE SPECIES	COMPOUND	Oxychlordane	C-Chlordane	T-Chlordane	C-Nonachlor	T-Nonachlor	Heptachlor	Heptachlor Ex.	Methoxychlor	PP'DDE	PP'DDD	PP'DDT	Dieldrin	Alpha BHC	Hexachlor	Tetradifon	Arochlor	ND = below detection limits								

Metal concentrations in soil from Great Dismal Swamp National Wildlife Refuge, August 1987. Table 9.

	3		Q	0.5	0.02	QN	3540	ND	QN	32.8	0.2	QN	ស	6.5	1300	97	7.4	ND	4	ហ	ND	0	QN	6.3	9.8	
	8		0.46	0.99	0.12	ND	21800	QN	ON	167	П	QN	17	7.6	9210	893	269	QN	8.2	35	QN	26.2	ND	22	76.2	
	PS4		0.2	1.7	0.084	ND	9290	ΩN	ND	93.1	0.57	ND	თ	4.6	6840	512	27	QN	4	28	QN	15	ND	14	32	
	PS3		0.4	3.3	0.04	QN	7890	ΩN	QN	51.4	0.37	ND	7.9	4.9	11400	399	21	QN	m	16	ND	9.8	ND	15	43.4	
ht).	PS2		Q	0.52	0.045	QN	3120	QN	ΩN	19.3	ND	NO	ស	2.3	8480	210	17	ND	7	σ	QN QN	5.1	QN	7.2	25	
(dry-weight).	PS1		ND	1.9	0.03	ND	6470	QN O	QN	27.9	QN	QN	7.7	3.2	12000	400	55.9	QN	m	10	NO	<b>&amp;</b>	Q.	16	20	
-million	RR		0.2	1.1	0.057	ND	8640	QN	QN	51.3	0.2	QN	7.4	6.2	4200	302	20	ND	ND	19	ON	9.3	QN	9.7	15	
parts-per-million	CIA.		0.68	1.1	0.17	ND	16600	QN	ON ON	138	1.1	QN	11	7.1	4530	501	34	QN	4	26	QN QN	20.5	QN	16	15	
are in	<b>DB</b> .		0.1	0.52	0.057	QN	0609	ND	ON	46.7	0.2	ND	6.5	3.7	2770	184	8.7	NO	ND	10	N	6.3	QN	9.3	11	
Concentrations	ED		0.2	2.5	0.054	ON C	4490	ND	ND	20.9	0.1	Q.	7.2	3.6	3160	194	5.9	ON	QN	14	ND	4.6	QN	σ	12	
Conc	SITE	COMPOUND	Se	As	Hq	Aq	Al	As	Ø	Ва	Be	g	Cr	ņ	F	Mq	W.	Mo	Ni	Pb	Se	Sr	TI	>	Zn	

SITE

= WASHINGTON DITCH ED = EAST DITCH
BD = BADGER DITCH
WD = WASHINGTON DITCH
RR = RAILROAD DITCH

PS = POCOSIN SWAMP CS = CYPRESS SWAMP LD = LAKE DRUMMOND

Organochlorine concentrations in soil from Great Dismal Swamp National Wildlife Refuge, August Concentrations are in parts-per-million (wet-weight). Table 10.

				_	_	_		_	а,	_		_	01	~	_	_	
	CS		0.00	0.00	0.001	0.00	0.00	N	0.00	N	0.004	N	0.00	0.003	N	N	
	PS4		0.001	0.001	0.001	QN QN	0.001	0.002	0.001	QN	0.005	QN	0.002	0.003	Q.	Q	
	PS3		0.001	0.001	0.001	QN	0.001	QN	QN	900.0	0.003	QN	0.003	0.003	0.002	ND	
weignt).	PS2		0.001	0.001	0.001	QN	0.001	NO	QN	QN	0.001	QN	0.002	QN	0.002	ND	
are in parts-per-million (wet-weight)	PS1		0.001	0.001	0.001	QN	0.001	NO	QN	N	0.002	QN	QN	QN	QN	ND	
r-mılııc	RR		NO	QN	QN	NO	ND	N	NO	QN	0.002	Q	0.002	0.004	ON	ND	
parts-pe	WD		ND	QN	QN	QN	QN	QN	QN	ND	0.001	QN	0.002	0.003	ND	ND	
are ın	BD		NO	0.001	0.001	QN	QN	QN ON	0.001	QN	0.002	QN	0.003	NO	ND	NO	
centrations	RD				ND						0.002	ND	0.001	0.002	ON	0.002	
198/. Concent	SITE	COMPOUND	Oxychlordane	C-Chlordane	T-Chlordane	C-Nonachlor	T-Nonachlor	Heptachlor	Hept. Epox.	Methoxychlor	PP'DDE	OP'DDD	PP'DDD	PP'DDT	Dieldrin	Alpha BHC	

SITE

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= POCOSIN SWAMP = CYPRESS SWAMP PS

Alkane concentrations in soil from the Great Dismal Swamp National Wildlife Refuge, August 1987. Concentrations are in parts-per-million (wet-weight). Table 11.

SITE	ED	ВД	QW.	RR	PS1	PS2	PS3	PS4	CS
COMPOUND									
n-Dodecane	0.35	0.32	0.31	0.37	0.35	0.15	0.21	0.27	0.24
n-Tridecane	QN	ND	QN	QN	QN	ND	QN	QN	ND
n-Tetradecane	QN	ND	QN	QN	ND	ND	QN	ND	ND
Octylcyclohexane	QN	ND	NO	ND	QN	ND	ND	QN	ON
n-Pentadecane	Q.	ND	QN	QN	ND	ND	ND	ND	ND
Nonylcyclohexane	Q	ON	N QN	ND	QN	QN	ON	ON	QN
n-Hexadecane	QN	ON	QN	ND	ND	ND	ND	ND	ND
n-Heptadecane	QN	ND	ND	ON	QN	QN	ND	QN	ND
Pristane	N N	ON	ND	0.12	ND	ON	NO	QN	QN
n Octadecane	QN	NO	QN	0.04	QN	QN	ON	QN	QN
Phytane	QN	QN	ND	QN	QN	QN	QN	QN	ON
n-Nonadecane	QN	0.21	ND	0.05	QN	QN	ND	QN	QN
n-Eicosane	QN	QN	0.05	0.05	ND	ΩN	QN	0.04	0.05
n-Heineicosane	Q	0.07	0.34	60.0	0.04	0.03	0.05	0.13	0.14

SITE

ED = EAST DITCH
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Polycylic aromatic hydrocarbon concentrations in soil from Great Dismal Swamp National Wildlife Refuge, August 1987. Concentrations are in parts-per-million (wet-weight). Table 12.

SITE	ED	BD	QW Qw	RR	PS1	l PS2	PS3	P.S.4	CS
COMPOUND									
Naphthalene	QN	QN	QN	90.0	ON		ND	QN	QN
Acenaphthylene	QN	QN	QN	QN	QN	ND	QN	ND	QN
Acenaphthene	ND	QN	ND	ND	NO		QN	ND	NO
Fluorene	Q	QN	ND	ND	QN		ON	QN	ND
Phenanthrene	90.0	90.0	NO	0.13	ON	0	0.09	0.07	90.0
Anthracene	S	QN	NO	0.12	ND		ND	QN	ND
Fluoranthene	QN	QN	QN	ND	QN		0.09	ND	ND
Pyrene	QN	QN	ND	QN	ND	0	0.11	QN	ND
Benzo(a)anthracene	QN	QN	ND	ND	QN		QN	ND	ND
Chrysene	ND	QN	ND	ND	ON		90.0	ND	ON
Benzo(b)fluoranthene	Q	QN	ND	ND	N		90.0	ON	ON
Benzo(k)fluoranthene	QN	QN	QN	ND	N		QN	ND	ND
Benzo(a)pyrene	QN	ND	ND	QN	QN		QN	ND	ND
Benzo(e)pyrene	Q	ND	ND	ND	N		QN	ND	N
Perylene	~	QN	ND	ND	QN		QN	ND	ND
Indeno(1,2,3-cd)pyrene	~	QN	ND	ND	NO		QN	ND	ND
Dibenz(ah)anthracene	~	QN	QN	ND	QN N		QN	QN	ND
Benzo(ghi)perylene	ND	QN	QN	ND	ND		ND	ND	QN

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RAILROAD DITCH

Table 13. Metal concentrations in sediments from Great Dismal Swamp National Wildlife Refuge, August 1987. Concentrations are in parts-per-million (dry-weight).

SITE	ED	BD	WD	RR	PS1	cs	MD	LD
COMPOUND	2							
Se	0.66	0.3	ND	1.2	0.2	0.79	0.82	0.83
As	24	0.67	0.3	1	1.2	3	4.3	1.3
Нg	0.13	0.045	0.03	0.072	0.054	0.15	0.081	0.03
Ag	ND	ND	ND	ND	ND	ND	ND	ND
Al	17400	9440	4480	11900	7150	20700	6100	2260
As	ND	ND	ND	ND	ND	ND	ND	ND
В	3	ND	ND	ND	ND	ND	ND	ND
Ba	73.7	76.3	30.8	104	68.8	184	56	31.4
Ве	0.93	0.47	0.2	1.4	0.71	1.2	0.5	0.3
Cd	2.2	ND	ND	ND	ND	0.7	ND	ND
Cr	19	8.9	4	8.6	6.2	16	5.4	3
Cu	42	3	1.7	12	3.5	9.8	8.6	5.3
Fe	84200	3560	1760	8100	5340	10600	7100	1610
Mg	1060	353	179	444	313	807	318	156
Mn	123	14	18	14	26	216	15	12
Mo	ND	ND	ND	ND	ND	ND	ND	ND
Ni	15	4	ND	4	4	9.3	4	4
Pb	120	10	9	17	17	44	10	8
Se	ND	ND	ND	ND	ND	ND	ND	ND
Sr	52.6	15.2	5.6	78.4	13.8	29.3	28.5	14.7
Tl	ND	ND	ND	ND	ND	ND	ND	ND
v	30	12	6.2	23	11	24	16	9.1
Zn	420	24	13	12	40.8	67.4	51	27

#### SITE

ED = EAST DITCH

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PS = POCOSIN SWAMP

MD = MIDDLE DITCH

BD = BADGER DITCH

RR = RAILROAD DITCH

CS = CYPRESS SWAMP

LD = LAKE DRUMMOND

Organochlorine concentrations in sediment from Great Dismal Swamp National Wildlife Refuge, Table 14.

r	August 1987. Concentrations are in parts-per-million (wet-weight).	Concent	rations	are in p	Concentrations are in parts-per-million (wet-weight).	Greac million	Wet-we	sight).	Tollar W	airini
	SITE	ED	BD	Q <sub>M</sub>	RR	PS1	S	Æ	LD1	LD2
	COMPOUND									
	Oxychlordane	QN	QN	ND	0.002	QN	0.003	QN	QN	ON
	C-Chlordane	0.002	0.001	0.002	0.001	0.001	0.002	0.001	QN	0.001
*	T-Chlordane	0.002	0.001	0.001	0.002	0.001	0.003	0.001	0.001	0.001
	C-Nonachlor	0.001	ND	0.002	QN	QN	0.002	QN	ON	QN
	T-Nonachlor	0.002	0.001	QN	ND	0.001	0.002	0.001	QN	QN
	Hept. Epox.	ND	ND	QN	0.001	Q	QN	0.001	ON	0.001
	Methoxychlor	QN	0.005	ND	ND	0.005	0.016	0.007	QN	ON
	PP'DDE	0.003	0.002	0.003	ND	0.002	0.004	0.002	0.001	0.002
	OP'DDD	QN	QN	Q	QN Q	ND	900.0	0.003	QN	ON
	PP'DDD	900.0	0.005	0.005	0.004	0.004	0.014	0.008	0.003	Q
	PP'DDT	QN	ND	Q	0.011	0.004	0.014	0.007	0.007	0.005
	Tetradifon	QN	ND	QN	NO	ND	ON	QN Q	0.003	QN

EAST DITCH

WASHINGTON DITCH BADGER DITCH

RAILROAD DITCH

POCOSIN SWAMP CYPRESS SWAMP MIDDLE DITCH

= LAKE DRUMMOND 

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Table 15. Alkane concentrations in sediment from the Great Dismal Swamp National Wildlife Refuge, August 1987. Concentrations are in parts-per million (wet-weight).

SITE	ED	ВО	Q.	RR	PS1	cs	W	LD1	LD2
COMPOUND									
n-Dodecane	0.24	0.36	0.32	0.25	0.31	0.29	0.31	0.27	0.32
n-Tridecane	ND	ND	QN	QN	ND	ND	QN	ND	ON
n-Tetradecane	ND	ND	QN	NO	ND	ND	ND	ND	NO
Octylcyclohexane	ND	ND	QN	ON	ND	ND	ND	ND	ON
n-Pentadecane	0.03	QN	ND	NO	ND	ND	ND	ND	ON
Nonylcyclohexane	ND	ND	QN	QN	QN	ND	ND	QN	QN
n-Hexadecane	0.07	0.05	90.0	90.0	ND	ND	ND	QN	QN
n-Heptadecane	ND	0.15	0.15	0.2	0.05	0.05	90.0	90.0	QN
Pristane	ND	0.1	0.07	0.08	ND	ND	ND	QN	N
n_Octadecane	0.13	0.15	0.18	0.19	0.05	ND	ND	ON	ON
Phytane	0.39	0.11	0.05	90.0	ND	ND	0.09	QN	ON
n-Nonadecane	0.2	0.12	0.08	0.24	0.05	0.12	0.09	0.3	0.04
n-Eicosane	0.23	0.07	90.0	0.08	0.05	90.0	0.05	0.04	ON
n-Heineicosane	0.33	0.13	0.12	0.3	0.13	0.16	0.14	0.22	0.05

SITE

ED = EAST DITCH
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WD = WASHINGTON DITCH RR = RAILROAD DITCH

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LD = LAKE DRUMMOND PS = POCOSIN SWAMP MD = MIDDLE DITCH CS = CYPRESS SWAMP

Polycyclic aromatic hydrocarbon concentrations in sediment from Great Dismal Swamp National Wildlife Refuge, August 1987. Concentrations are in parts-per-million (wet-weight). Table 16.

SITE	ED	BD	WD	RR	PS1	CS	W	LD1	LD2
COMPOUND									
Naphthalene	ND	ND	ND	ND	S	QN	QN	QN	8
ne	ND	ND	NO	QN	ND	ND	ND	QN	ND
hene	ND	N	ND	ND	NO	ND	ND	QN	QN
	NO	ND	ΩN	QN	QN	ND	ON	ND	S
ne	0.54	0.11	Ω	90.0	90.0	90.0	60.0	ON	90.0
	NO	0.11	NΩ	QN	ND	QN	ND	ON	QN
nthene	1.85	N	ND	ND	ND	QN	0.08	NO	S
Pyrene	1.18	ND	QN	QN	ND	QN	90.0	QN	QN
anthracene	0.08	N	ND	QN	ND	QN	QN	QN	QN
	0.47	N	ND	ND	ND	NO	QN QN	WD	Q
	0.22	NO	NO	ND	QN	QN	Q	Q.	Q
nthene	0.16	NO	ND	NO	ND	ND	QN	ON	S
	90.0	ND	NO	ND	ND	ND	NO	ND	QN
Benzo(e)pyrene	0.13	QN	QN	QN	NO	ND	ON	ND	QN
	N	QN	ND	0.36	ND	ND	QN	ND	N
Indeno(1,2,3-cd)pyrene	ND	ND	N	ND	QN	ND	ON	ND	Q
	Q Q	NO	ON	ΩN	NO	ND	QN	ND	B
Benzo(ghi)perylene	ND	NO	ND	QN	ND	QN	ND	Q.	QN

ED = EAST DITCH
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RR = RAILROAD DITCH PS = POCOSIN SWAMP

CS = CYPRESS SWAMP
MD = MIDDLE DITCH

= LAKE DRUMMOND

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feta.	part	
7. 1	1	
Table 17. Metals concentrations in water fro		
Tab		

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PS1 PS4
3
el el

PS = POCOSIN SWAMP
JD = JERICHO DITCH
ED = EAST DITCH
LD = LAKE DRUMMOND
HP = HALL POCOSIN SWAMP

CC = CROSS CANAL
CD = CORAPEAKE DITCH
MS = MOSS SWAMP
RR = RAILROAD DITCH
WD = WASHINGTON DITCH

Metal concentrations in surface water from Great Dismal Swamp National Wildlife Refuge, July 1989. Concentrations are in parts per million (ppm). Table 18.

	3		.001	.001	ð	.004	2	1.25	9	.052	B	B	B	2	B	4.06	£	1.5	.048	2	2	S	.059	B	B	2	.025	
	302		.002	.002	Ø	.003	B	1.01	NO	.033	Q	.026	S	NO	S S	m	Q	1.75	.021	Q.	Q	2	.04	S	8	2	.026	
	ED		.004	.001	ND	800.	QN Q	1.18	Q	.054	ND	.038	Q	Q.	g	2.88	Q.	1.65	.032	Q.	Ð	ğ	.058	N	Q	B	.02	
	<b>10</b>			.002																								
	PS7			QN																								
	PS7			.001																								
	PS6			.001																								
	PSS			.001																								
	PS4			Ñ.																								
	PS3			N O																								
	PS2			£																								
	PS1			R																								
	WD			Q																								
	S			.002																								
i i	RR																										.022	
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	H	ļ	$\sim$ 1	. E	2	600.	S	5.46	N	.083	N	760	IN	Z	10.	6.2	2		20.0	2	, Z	; 2	0.4	2	. 2	. 2	.049	
	SITE	Ш	COMPOUNI	n 0	3 3	P Q	HG	[ A	q.	B	3 8	) ac	i Ü	1 2	3 8	φ Φ	, 4 , 4	a t	, X	X	) <u>:</u>	1 4	r v	Ē	י ני	>	Zn	

WD = WASHINGTON DITCH PS = POCOSIN SWAMP JD = JERICHO DITCH ED = EAST DITCH LD = LAKE DRUMMOND

CC = CROSS CANAL
CD = CORAPEAKE DITCH
MS = MOSS SWAMP
RR = RAILROAD DITCH
CS = CYPRESS SWAMP
HP = HALL POCOSIN SWAMP

SILES

ND = below detection limit

#### APPENDIX A

#### MAMMALS FOUND IN GREAT DISMAL SWAMP

Opossum (<u>Didelphis</u> <u>marsupialis</u>)

Dismal Swamp Southeastern Shrew (Sorex longirostris fisheri)

Dismal Swamp Short-tailed Shrew (Blarina telmalestes)

Least Shrew (Cryptotis parva)

Common Mole (Scalopus aquaticus)

Star-nosed Mole (Condylura cristata)

Keen Bat (Myotis keenii)

Pipistrelle (Pipistrellus subflavus)

Red Bat (Lasiurus borealis)

Evening Bat (Nycticeius humeralis)

LeConte's Big-eared Bat (Corynorhinus macrotis)

Cottontail (Sylvilagus floridanus)

Marsh Rabbit (Sylvilagus palustris)

Eastern Chipmunk (Tamias striatus)

Gray Squirrel (Sciurus carolinensis)

Southern Flying Squirrel (Glaucomys volans)

Rice Rat (Oryzomys palustris)

Harvest Mouse (Reithrodontomys humulis)

White-footed Mouse (Peromyscus leucopus)

Cotton Mouse (Peromyscus gossypinus)

Golden Mouse (Peromyscus nuttalli)

Lemming Mouse (Synaptomys cooperi)

Meadow Vole (Microtus pennsylvanicus)

Muskrat (Ondatra zibethicus)

Gray Fox (<u>Urocyon cinereoargenteus</u>)

Black Bear (Euarctos americanus)

Raccoon (Procyon lotor)

Longtail Weasel (Mustela frenata)

Mink (Mustela vison)

Otter (<u>Lutra canadensis</u>)

Bobcat (Lynx rufus)

White-tailed Deer (Odocoileus virginianus)

Source: Great Dismal Swamp National Wildlife Refuge.

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#### APPENDIX B

#### FISH SPECIES IN GREAT DISMAL SWAMP

Longnose Gar (Lepisosteus ossens)

Bowfin (Amia calva)

Redfin Pickerel (Esox americanus)

Chain Pickerel (Esox niger)

Golden Shiner (Notemigonus crysoleucas)

White Catfish (Ictalurus catus)

Channel Catfish (Ictalurus punctatus)

Yellow Bullhead (Ictalurus natalis)

Brown Bullhead (<u>Ictalurus nebulosus</u>)

American Eel (Anguilla rostrata)

Mosquitofish (Gambusia affinis)

Swampfish (Chologaster cornuta)

Pirate Perch (Aphredoderus sayanus)

Mud Sunfish (Acantharchus pomotis)

Flier (Centrarchus macropterus)

Warmouth (Chaenobryttus gulosus)

Blue Spotted Sunfish (Enneacanthus gloriosus)

Banded Sunfish (Enneacanthus obesus)

Redbreast Sunfish (Lepomis auritus)

Pumpkin Seed (Lepomis gibbosus)

Bluegill (Lepomis macrochirus)

Largemouth Bass (Micropterus salmoides)

Black Crappie (Pomoxis nigremaculatus)

Swamp Darter (<a href="Etheostoma fusiforme">Etheostoma fusiforme</a>)

Yellow Perch (Perca flavescens)

Eastern Mudminnow (<u>Umbra pygmaea</u>)

Creek Chubsucker (Erimyzon oblongus)

Common, scientific, and family names are from Robins et al. 1991.

(Source: Great Dismal Swamp National Wildlife Refuge)

## APPENDIX C

COMPOUNDS ANALYZED

AND

METHODS OF ANALYSIS

Aluminum (Al)

Arsenic (As)

Antimony (Sb)

Barium (Ba)

Beryllium (Be)

Boron (B)

Cadmium (Cd)

Chromium (Cr)

Copper (Cu)

Iron (Fe)

Lead (Pb)

Magnesium (Mg)

Manganese (Mn)

Mercury (Hg)

Molybdenum (Mo)

Nickel (Ni)

Selenium (Se)

Silver (Ag)

Strontium (Sr)

Thallium (T1)

Tin (Sn)

Vanadium (V)

Zinc (Zn)

# Alkane Compounds Analyzed

n-Dodecane
n-Tridecane
n-Tetradecane
Octylcyclohexane
n-Pentadecane
Nonylcyclohexane
n-Hexadecane
n-Heptadecane
Pristane
n-Octadecane
Phytane
n-Nonadecane
n-Eicosane
n-Heineicosane

Organophosphate and Carbamate Compounds Analyzed

Organophosphate Compounds Analyzed

R = Trade Name

Acephate

69

Azinphos-methyl

Chlorpyrifos-dursban<sup>R</sup>

Coumaphos

Demeton

Diazinon

Dichlorvos

Dicrotophos

Dimethoate

Disulfoton

Dursban<sup>R</sup>

EPN (O-Ethyl O(4-nitrophenyl) phenylphosphonothioate)

Ethoprop

Famphur

Fensulfothion

Fenthion

Malathion

Methamidophos

Methyl Parathion

Mevinphos

Monocrotophos

Parathion

Phorate

Terbufos

Trichlorfon

Sulprofos

Trichloronate

Carbamate Compounds Analyzed

Aldicarb

Oxamyl

Carbaryl

Methomyl

Carbofuran

Methiocarb

### Organochlorine Compounds Analyzed

```
Arochlor<sup>R</sup> 1232
HCB (Hexachlorobenzene)
                                               Arochlor<sup>R</sup> 1242
\alpha-BHC (benzene hexachloride)
                                               Arochlor<sup>R</sup> 1248
Γ-BHC (benzene hexachloride)
                                               Arochlor<sup>R</sup> 1254
\beta-BHC (benzene hexachloride)
                                               Arochlor<sup>R</sup> 1260
\delta-BHC (benzene hexachloride)
                                               Arochlor<sup>R</sup> 1016
Oxychlordane
                                              R = Trade Name
Heptachlor
Heptachlor Epoxide
Methoxychlor
c-Chlordane
t-Nonachlor
Toxaphene
PCBs (Polychlorinated biphenyls (total))
o, p'-DDE (dichlorodiphenyldichloroethylene)
t-Chlordane
p, p'-DDE (dichlorodiphenyldichloroethylene)
Dieldrin
Aldrin
o, p'-DDD (1,1-dichloro-2,2-bis(p-chlorophenyl) ethanex)
Endrin
c-nonachlor
o, p'-DDT (dichlorodiphenyltrichloroethane)
p, p'-DDD (1,1-dichloro-2,2-bis(p-chlorophenyl) ethanex)
p, p'-DDT (dichlorodiphenyltrichloroethane)
Mirex
Endosulfan I
Endosulfan II
Endosulfan sulfate
DCPA (Dimethyl tetrachloroterephthalate)
Dicofol
Tetradifon
Arochlor<sup>R</sup> 1221
```

# Polyaromatic Hydrocarbons Analyzed

Naphthalene

Acenaphthylene

Acenaphthene

Fluorene

Phenanthrene

Anthracene

Fluoranthene

Pyrene

Benzo(a) anthracene

Chrysene

Benzo(b) fluoranthene

Benzo(k) fluoranthene

Benzo(a)pyrene

Benzo(e)pyrene

Perylene

Indeno(1,2,3-cd)pyrene

Dibenz (ah) anghracene

Benzo(ghi)perylene



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#### % MOISTURE

For animal tissue and sediments of sufficient size, moisture was determined by placing a weighed aliquot of the sample in a Fisher Isotemp oven and drying at 103-105°C. The dried sample was then weighed and the data entered into a computer program to generate the % moisture and final report.

Plants, and samples too small for oven dried moisture determination had the % moisture calculated from the moisture lost during the freeze-drying in the Labcono Freeze-Dryer 8. The data was entered into a computer program to generate a % moisture and final report.



#### HOMOGENIZATION

Large tissue samples, such as whole fish, were first run through a meat grinder one or more times depending on the size of the sample. An aliquot of the ground sample was weighed and frozen. For smaller tissue samples and plant samples the entire sample was weighed and then frozen. For sediments, the sample was mixed and an aliquot weighed and frozen. The frozen samples were placed in a Labcono Freeze Dryer 8 until the moisture had been removed. The dry samples were then weighed and further homogenized using a blender, or Spex Industries, Inc. Model 8000 mixer/mill with tungsten-carbide vial and balls.



## NITRIC - PERCHOLORIC DIGESTION - (ICP)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. quartz Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly 15 ml. of concentrated sub-boiled  ${\sf HNO}_3$  and 2.5 ml. of concentrated sub-boiled  ${\sf HC1O}_4$ were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the  $\ensuremath{\mathsf{HNO}}_3$  began refluxing, samples were allowed to reflux overnight. (This decreased the chance for charring during the reaction with  ${
m HC10}_4$ .) After the refluxing, the heat was gradually increased until the  ${\rm HNO}_3$  had been driven off, and the reaction with HClO<sub>4</sub> had occured. When dense white fumes from the  ${
m HC10}_4$  were evident, the samples were removed from the heat and allowed to cool. Two ml. of concentrated sub-boiled HC1 were added. The flasks were replaced on the heat and warmed until the containers were hot to the touch or started to boil. They were removed from the heat, and 5-10 ml. of deionized water were added. Samples were allowed to cool. They were then diluted using deionized water in a 50 ml. volumetric flask and transferred to clean, labeled, 2 oz. polyethylene bottles.



## NITRIC REFLUX DIGESTION FOR MERCURY

Approximately 0.5 g. of sample was weighed into a freshly cleaned 50 ml. round bottom flask with 24/40 ground glass neck. For waters, 10 ml. of sample were measured into the flask. Five ml. of concentrated sub-boiled  $\mathrm{HNO}_3$  were added and the flask was placed under a 12 inch water-cooled condenser with water running through the condenser. The heat was turned up to allow the  $\mathrm{HNO}_3$  to reflux no more than 1/3 the height of the columns. Samples were allowed to reflux for two hours. Then the heat was turned off and the samples allowed to cool. The condensers were rinsed with 1% v/v HCl and the flasks removed. The samples were diluted with 1% v/v HCl in a 50 ml. volumetric flask and then transferred to clean, labeled, 2 oz. flint glass bottles.



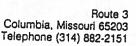
## NITRIC - PERCHOLORIC DIGESTION - (SELENIUM)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. quartz Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon breakers.) For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly 15 ml. of concentrated sub-boiled  ${
m HNO_3}$  and 2.5 ml. of concentrated sub-boiled  ${
m HC1O_4}$ were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the  $\ensuremath{\mathsf{HNO}}_3$  began refluxing, samples were allowed to reflux overnight. (This decreased the chance for charring during the reaction with  ${
m HC10}_4$ .) After the refluxing, the heat was gradually increased until the  ${
m HNO}_3$  had been driven off, and the reaction with  ${
m HC10}_4$  had occured. When dense white fumes from the  ${
m HC10}_4$  were evident, the samples were removed from the heat and allowed to cool. Two ml. of concentrated sub-boiled HCl were added. flasks were replaced on the heat and warmed until the containers were hot to the touch or started to boil. They were removed from the heat, and 5-10 ml. of deionized water were added. Samples were allowed to cool. They were then diluted using deionized water in a 50 ml. volumetric flask and transferred to clean, labeled, 2 oz. polyethylene bottles.



## NITRIC - PERCHOLORIC DIGESTION - (ARSENIC)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly 15 ml. of concentrated sub-boiled  $\mathrm{HNO_3}$  and 2.5 ml. of concentrated sub-boiled  $\mathrm{HClO_4}$  were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the  $\mathrm{HNO_3}$  had been driven off, and the reaction with  $\mathrm{HClO_4}$  had occured. After this reaction, the samples were heated approximately 5 minutes, after dense white fumes from the  $\mathrm{HClO_4}$  were evident. The samples were removed from the heat and allowed to cool. Samples were diluted using deionized water in 50 ml. volumetric flasks and transferred to clean, labeled, 2 oz. polyethylene bottles.





## INDUCTIVELY COUPLED PLASMA (ICP)

The instrument used for ICP analysis was a Jarrell-Ash Model 1100 Mark III with 40 analytical channels, controlled by a Digital Equipment Company (DEC) 11/23+ computer with two RLO2 disk drives, DEC VT100 terminal, and DEC LA120 decwriter III. The instrument was standardized with a series of seven standards containing 36 elements. After the standardization, the detection limit was determined by taking ten integrations of the zero standard; three times the standard deviation of the mean was used as the detection limit. Instrumental quality control samples were then analyzed to check the ICP operation. If the values were acceptable, the samples were then analyzed. Standards were run every 10-15 samples to check for drift. If the drift was more than 5%, the instrument was restandardized. After the analyses were completed, the data were transferred to the Perkin-Elmer LIMS 2000 computer for calculation. The final detection limit for each element was further increased by 4% of the magnitude of the spectral interferences from the other elements. The data were checked before calculation to correct for possible errors in sample number, weight, volumes and dilution. The data were calculated using the ICP calculation program written by ETSRC computer staff, which corrected for blanks, standard drift, spectral interferences, sample weight, sample volume, and dilution. After the quality control was reviewed, a final report was generated using a Hewlett-Packard laser jet printer.

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## MERCURY - COLD VAPOR ATOMIC ABSORPTION

Equipment used for Cold Vapor Atomic Absorption include: Perkin-Elmer Model 403 AA; Perkin-Elmer Model 056 recorder; Technicon Sampler I; Technicon Pump II; a glass cell with quartz windows and capillary tube for entry and exit of the mercury vapor; and a liquid-gas separator. The samples were placed in 4 ml. sample cups at least 3/4 full. The samples were mixed with hydroxylamine for preliminary reduction, then stannous chloride for reduction to the mercury vapor. The vapor was separated from the liquid and passed through the cell mounted in the light path of the burner compartment. The peaks were recorded and the peak heights measured. The standardization was done with at least 5 standards in the range of 0 to 10 ppb. The correlation coefficient was usually 0.9999 or better and must have been at least 0.999 to have been acceptable. A standard was run every 8-10 samples to check for drift in the standardization. This was usually less than 5%. Standards were preserved with 10% v/v HNO3, 1% v/v HCl and 0.05% w/v K2Cr2O7. The solution concentrations were calculated and the data entered into the AA calculation program which corrected for blank, dilution, sample weight, sample volume and entered the data into the LIMS system for report generation.



### ARSENIC AND SELENIUM BY HYDRIDE

The Varian VGA-76 hydride generation accessory was mounted on either a Perkin-Elmer Model 603 AA or Model 3030 (B) AA. Electrodeless Discharge lamps (EDL) were used. The instrument and EDL settings were taken from the instrument manuals. The burner mount for a Perkin-Elmer Model 10 Hydride generator was modified slightly to hold the Varian quartz cell. The cell was aligned in the light path of the burner chamber and a very lean flame was used for heating the cell. The two stock solutions were 50% v/v sub-boiled HCl and 0.6%  $NaBH_4$  in 0.5% NaOH for Selenium and concentrated sub-boiled HCL and 1%  $NaBH_4$  in 0.5% NaOH for Arsenic. Samples were diluted with 10% v/v sub-boiled HCl. Standards were prepared by dilution of Fisher 1000 ppm stock with 10% v/v sub-boiled HCl in the range of 0 to 20 PPB. The instrument was standardized to read directly in PPB using S1 = 5.00 and S2 = 20.00. After standardization, the standardization was checked by reading other standards such as 2.00, 10.00 and 15.00 PPB and an instrumental quality control sample with a known value. If the standards and quality control were acceptable, the detection limit was determined by reading the zero standard 10 times, and twice the standard deviation of the mean was used as the detection limit. Samples were analyzed by taking an integrated reading for 3 seconds after the plateau was reached for the sample. This occured approximately 45 seconds after the sample tube was placed in the sample. Standardization was checked every 8-15 samples and approximately 10% of the samples were checked by the method of additions to monitor matrix effects. Matrix effects were usually not significant with the VGA-76. The data was corrected for drift of the standard curve and entered into the AA calculation program. This program corrected for blank, dilution, sample weight, sample volume and recorded the data in the LIMS database for report generation.



## ARSENIC IN FISH AND MARINE SAMPLES BY HYDRIDE

The Perkin-Elmer MHS-1 hydride generation accessory was mounted on either a Perkin-Elmer Model 603 AA or Model 3030(B) AA. An Electrodeless Discharge Lamp (EDL) was used. The instrument and EDL settings were taken from the instrument manuals. The cell was aligned in the light path of the burner chamber and a very lean flame used for heating the cell. The standard curve was run and a quality control sample of known concentration was then run to check the standard curve. Blanks and samples are run by diluting an aliquot of the digested sample to 10 ml. with 4% v/v HClO<sub>4</sub>. The amount of sample used varied with the Arsenic concentration. Samples were analyzed using the method of standard additions. The peaks, from the recorder tracing, were measured with a ruler and the slope and intercept calculated on a calculator. The data were entered into the AA calculation program. This program corrected for the blank, dilution factors, sample weight, sample volume and recorded the data in the LIMS database for report generation.

## ANALYTICAL METHODOLOGY FOR ORGANOPHOSPHATE/CARBAMATE SCANNING

#### CATALOG 5912

MATRIX - WATER SAMPLE PREPARATION DATE - 8/7/89 COMPLETION DATE - 8/31/89

SUMMARY - The extraction procedure was a modified version of the one described in the EPA test Method #608, Section 10 (1). The water samples were extracted three times with methylene chloride. The methylene chloride extracts were combined, dried with sodium sulfate and concentrated on a rotary evaporator. Sample extracts were refrigerated prior to analysis. Residues were quantified by gas chromatography using either an instrument selective for organophosphate pesticides (Flame Photometric Detector) or one selective for carbamate pesticides (Nitrogen Phosphorous Detector) similar to Belisle et al (1988) (2). Megabore capillary columns were used for the GC separation.

#### DEVIATIONS -

a. From Belisle et. al.: 30 m megabore 7% cyanopropyl 7 % phenyl polysiloxane (film thickness 1.0 micron) was used for organophosphate pesticides. 30 m megabore 5% phenyl methyl polysiloxane (film thickness 1.5 micron) was used for carbamate pesticides. Temperature program for OPs: 150°C, hold for 5 min.; rate 10°C/min to 240°C, hold for 22 min. Temperature program for carbamates: 100°C, hold for 2 min; rate 5°C/min to 180°C, hold for 2 min; 20°C/min to 240°C, hold for 17 min.

PROBLEMS ENCOUNTERED - Samples arrived frozen on 8/4/89. Three samples (16DU68, 150P66, and 120P68) were not analyzed due to broken jars. Samples 080P66 and 090P66 were cracked but appeared to be OK. After thawing all samples (including the ones in the non broken jars) were immediately transferred with acetone into chemically cleaned jars since all samples needed to be treated in the same manner. The spiked samples were spiked on 8/4/89 and then extracted on 8/7/89. All jars were solvent rinsed except for the first sample of the duplicate pair. The rinsings from sample 020P65 were all transferred to the second sample of the duplicate.

#### GREAT DISMAL SWAMP SUFFOLK LANDFILL BATCH R5-87-013, CATALOG #5407 ORGANOCHLORINE AND AROCHLOR ANALYSIS

#### SAMPLE PREPARATION AND EXTRACTION

Eighteen soils, 14 fish and 9 small mammal samples were analyzed by Patuxent methods. The soil samples were homogenized. Each of the fish and mammal samples was minced on a high molecular weight polyethylene chopping block and homogenized in a food blender.

A subsample of the homogenate (2.6 g to 11.2 g), sodium sulfate (heat treated at 550°C), and internal standards were blended in a one-half-pint food blender. This mixture was added to a fiber extraction thimble (pre-extracted with petroleum ether) and extracted with petroleum ether (B&J distilled in glass) for at least 20 hours. The extract was concentrated to 10 mL with a Kuderna-Danish on a steam bath. At this point a 1 mL portion of the sample extract was removed for lipid determination (for tissue samples only). During the concentration stages, the extract was never allowed to go to dryness.

The 9 mL of extract was exchanged into methylene chloride (Omnisolve distilled in glass) and brought to a 10 mL volume. A volume of extract equivalent to approximately 1 g of sample was loaded into a loop on the GPC unit (ABC model No. 1002A) and injected. The GPC unit transfers the eluted fraction containing the chlorinated organics to an autoconcentrator that concentrates during elution and exchanges the solvent to hexane for a final volume of 10 mL.

The sample was concentrated to 1 mL by nitrogen blowdown and subjected to alumina micro column cleanup. The alumina (Biorad neutral alumina AG7, 100 to 200 mesh) was ignited at 550°C and then deactivated with distilled water (7% by weight). The analytes were eluted with 10 mL of 4:1 hexane/methylene chloride. The eluent was concentrated to 1 mL for GC capillary analysis. Sediments and soils had 50 uL of mercury added to the extract for sulfur removal.

#### LIPID AND MOISTURE DETERMINATION

The 1 mL of extract removed before the GPC step was used for lipid determination. The extract was dried at room temperature in tared aluminum pans to constant weight.

For moisture determination, 2 g of the tissue homogenate or mixed sediment was placed into a tared aluminum pan and placed in a drying oven (105°C) for at least 48 hours. The weight was recorded after cooling in a desiccator overnight.

#### INTERNAL STANDARDS

For organochlorine analysis, six chlorinated biphenyl congeners were added before extraction of the sample and served the following purposes:

1. Monitoring sample extract losses due to extraction efficiency, GPC cleanup, or extract transfer.

- 2. Estimating detection limits.
- 3. Increasing accuracy of predicted retention times  $(\pm 0.005 \text{ min})$  for the analytes.
- 4. Providing backup internal standards in the event of sample matrix interference with the normal quantification internal standard.

Before organochlorine GC analysis, two additional internal standards were added to the sample. These were used for monitoring the instrument's health; e.g., to indicate if there were any problems with the injection of each sample.

#### GC ANALYSIS

A Hewlett-Packard 5880A GC equipped with dual capillary column/dual ECD detectors was used for the organochlorine and arochlor analysis. The analysis was a single splitless (Grob) injection onto two 30-meter columns (DB-1 and DB-1701) of different polarities. The dual column analysis, besides providing confirmation of the pesticides, checks for coelution of unknowns with each individual Because of the high resolving power of the capillary columns, pesticide. coelution by an unknown on both columns was improbable. Except as explained below, the amount and variance shown on the sample report pages was calculated from the values given by the two GC columns for each compound detected. If the variance was greater than 15% of the mean, it was assumed that coelution was occurring on the column showing the higher amount and only the lower amount was reported. In that case, a variance indicator NA (Not Applicable) was printed in "Variance" list. Also, if near coelution occurs, where a positive identification on one of the GC columns was not possible, then only the amount given by the GC column that allows positive identification was reported. In this case, the variance indicator NA also was printed. The indicator NA also was used in the "Variance" list in cases where nothing was found above the detection limits on either column where the indicator ND was printed in the "Amount" list.

The temperature program was 50°C for two minutes to 280°C at 3°C/minute and a post-run temperature of 290°C for five minutes. Linear flow rate was 30 cm helium/second.

#### DATA ANALYSIS

Quantitation was done on the Hewlett-Packard 5880A GC. Due to the narrowness of the capillary peaks, all data were based on peak height, resulting in less biasing due to tailing, near coelution and baseline drift ("Assessment of the Results from Data Processing Systems using a Digital Chromatogram Simulator", R.J. Hunt, Journal of High Resolution Chromatography Communications, Vol. 8, July 1985, pp. 347-355). All data were collected directly from the GC into databases in an Amiga computer. The databases, besides providing report generation, allow the monitoring of the standard curves and internal standards over time. The data on the Amiga also were used for pattern recognition in arochlor analysis and to develop the organochlorine pesticide "unknowns" report. Appendices A and B contain the results of the organochlorine-arochlor and "unknowns" analyses, respectively.

#### BLANKS AND DUPLICATES

The batch size for soxhlet extraction was 12 (11 samples and 1 blank). Two batches went onto the GPC at a time. No analytes were detected in the blank at concentrations greater than 0.5 ppb.

Samples BD-FL-01C, WD-FL-01C, WD-GS-01C and LD-BF-01 were analyzed in duplicate. The following table summarizes these results.

		BD-FL-01C		
	<u>A</u>	<u>B</u>	Mean	Variance
PP'DDE	0.010	0.006	0.008	0.002
		WD-FL-01C		
PP'DDE	0.010	0.010	0.010	0.000
		WD-GS-01C		
PP'DDE PP'DDD PP'DDT	0.013 0.006 0.005	0.016 0.009 0.013	0.015 0.008 0.009	0.002 0.002 0.004
Į. X		LD-BF-01		
PP'DDE PP'DDD Arochlor 1260	0.041 0.029 0.044	0.042 0.027 0.049	0.042 0.028 0.047	0.001 0.001 0.003

#### GC/MS CONFIRMATION

GC/MS confirmation was done on sample ED-FL-01C containing the PP'DDE, PP'DDD and PP'DDT, which were confirmed. No other samples contained analytes greater than  $0.1~\rm ppm$ , which is the GC/MS detection limit.

#### **UNKNOWNS**

No unknowns were detected at or above the 0.09-ppm level. This report contains no Appendix B.

#### ALKANE AND AROMATIC ANALYSIS

#### SAMPLE PREPARATION

Sample preparation for the alkanes and aromatics was as follows. Five micrograms deuterium labeled surrogate spikes were added to 5-15 g of the sediment homogenate. There were labeled analogs for each of the polyaromatic hydrocarbons to be analyzed except benzo(e)pyrene and perylene. The sediments were mixed with sodium sulfate and soxhlet extracted overnight with methylene chloride. The combined organic extract filtered through muffled Na<sub>2</sub>SO<sub>4</sub> and rotary-evaporated to several milliliters. One hundred mL petroleum ether and 0.7 mL isooctane was added prior to initial evaporation and the extract again reduced to several milliliters.

The alkanes and aromatics were fractionated on a column of 20 g 2.0% water-deactivated silica gel. Alkanes were eluted with 110 mL petroleum ether. Aromatics were eluted with 100 mL 40% methylene chloride in petroleum ether and an additional 60 mL methylene chloride. Each fraction was concentrated by rotary evaporation followed by nitrogen evaporation. The alkane fraction was evaporated to 1 mL, internal standards added and the extract transferred to a vial in preparation for GC analysis.

The aromatic fraction was concentrated to 10 mL and cleaned by gel permeation chromatography on Bio-Beads SX-3. The collected gel permeation fraction was first rotary-evaporated, then nitrogen-evaporated to 1 mL and finally shaken with aqueous sodium hydroxide. This step removed residual fatty acids. An injection internal standard was added to each extract and it was transferred to a vial in preparation for GC analysis.

#### ALKANE INTERNAL STANDARDS

Three compounds, n-undecane, n-docosane, and n-triacontane were added to each of the final alkane extracts before GC analysis to serve as quantitation internal standards.

#### ALKANE GAS CHROMATOGRAPHY

Gas chromatography was done using a 30 M DB-5 capillary column with splitless injection on a Hewlett-Packard 5880A GC with flame ionization. The temperature program was 60°C for three minutes to 310°C at 6°/minute for alkanes and a post run temperature of 320°C for two minutes. Linear flow rate was 30 cm helium/second.

#### POLYAROMATIC HYDROCARBON INTERNAL STANDARDS

Internal standards for the polyaromatic hydrocarbons were the deuterium labeled compounds added at the saponification stage. The deuterium labeled fluorene has been found to deuterium/hydrogen exchange during base hydrolysis. Thus,  $D_{10}$  phenanthrene was used as the internal standard for fluorene.

Use of these internal standards automatically compensates for any losses during sample preparation. An injection internal standard was added to each extract before analysis on the GC/MS and was used to determine if recovery of labeled compounds were within the normal expected range.

#### POLYAROMATIC HYDROCARBON GAS CHROMATOGRAPHY/MASS SPECTROMETRY

Gas chromatography was done using a 30 M DB-5 capillary column with splitless injection on a Hewlett-Packard 5890 GC in conjunction with a Finnigan-MAT INCOS 50 mass spectrometer. The temperature program was 50°C for two minutes to 320°C at 8°/minute. The mass spectrometer scanned from 35 to 450 m/z in 0.56 seconds at 70 eV.

The target polyaromatic hydrocarbons were purchased from Supelco (Supelpreme) and mixtures of isotope labeled compounds were purchased from MSD Isotopes. Responses of the labeled compounds to 2,2'-difluorobiphenyl internal standard and of the target to the labeled compounds was used to create a polyaromatic hydrocarbon library response list. The response curves for the target polyaromatic hydrocarbons were generated from 1 to 50 ng on column and were linear in this range.

The mass spectrometer was calibrated and an on-going calibration verification standard at either 1 or 2 ng on column injected daily. Compounds were searched for and quantified with "TCA", a program available from Finnigan-MAT for the analysis of target compounds. Mass spectra were examined manually to verify identification.

Results of the alkane and polyaromatic hydrocarbon analyses are contained in Appendices C and D, respectively.

Method 5. Analysis For Aliphatic and Polynuclear Aromatic
Hydrocarbons and Organochlorine Pesticides In Water

A 500 milliliter water sample is extracted four times by shaking with 50 milliliter portions of methylene chloride. extracts are combined and concentrated by Kuderna-Danish to near dryness, then reconstituted in 5 milliliters petroleum ether. An appropriate aliquot is removed for organochlorine and PCB analysis and transfered to a 20 gram Florisil column. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction Fraction II is concentrated to appropriate volume for quantification of residues by packed or capillary column electron capture gas chromotography. Fraction I is concentrated and transferred to a silicic acid chromatographic column for additional cleanup required for separation of PCBs from other organochlorines. Three fractions are eluted from the silicic acid column. concentrated to appropriate volume for quantification of residues by packed or megabore column, electron capture gas chromotography. The remainder of the petroleum PCBs are found in Fraction II. ether from the above methylene chloride extraction is transferred to a 20 gram 1% deactivated silica gel column, topped with 5 grams neutral alumina. Aliphatic and polynuclear aromatic hydrocarbon residues are separated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) folllowed by elution of aromatics using first, 100 ml 40% methylene chloride/60% petroleum ether then 50 ml methylene chloride (combined eluates, Fraction If needed, Fraction I containing aliphatics is subjected to II).

Method 5. Continued

additional cleanup by concentration and transfer to a deactivated (2% water) Florisil column. Aliphatic residues are eluted from the Florisil column using 200 ml 6% diethyl ether/94% petroleum ether. The eluate is concentrated to appropriate volume for quantification by capillary column, flame ionization gas chromotography. The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride, and subjected to gel permeation chromatographic (GPC) cleanup prior to quantification by capillary, flame ionization gas chromatography and fluorescence HPLC.

# Elution Profiles for Florisil, Silica Gel and Silicic Acid Column Separations

#### A. Florisil Column:

Fraction I (6% ethyl ether containing 2% ethanol, 94% petroleum ether)

HCB, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordane, heptachlor epoxide, gamma-chlordane, trans-nonachlor, toxaphene, PCB's, o,p'-DDE, alpha-Chlordane, p,p'-DDE, p,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, mirex, dicofol, endosulfan I (Split with FII).

- 2. <u>Fraction II</u> (15% ethyl ether containing 2% ethanol, 85% petroleum ether)
  dieldrin, endrin, dacthal, endosulfan I (split with FI), endosulfan II (split with FIII), endosulfan sulfate (split with FIII).
- 3. Fraction III (50% ethyl ether containing 2% ethanol, 50% petroleum ether) endosulfan II (split with FII), endosulfan sulfate (split with FII), malathion.

## B. Florisil Mini-Column:

 Fraction I (12 ml hexane followed by 12 ml 1% methanol in hexane)

HCB, gamma-BHC (25%), alpha-BHC (splits with FII), trans-nonachlor, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD (splits with FII), o,p'-DDT, p,p'-DDT, mirex, cis-nonachlor, cis-chlordane, trans-chlordane, PCB's, Photomirex and derivatives.

2. Fraction II (24 ml 1% methanol in hexane) gamma BHC (75%), beta-BHC, alpha-BHC (splits with FI), delta-BHC, oxychlordane, heptachlor epoxide, toxaphene, dicofol, dacthal, endosulfan I, endosulfan II, endosulfan sulfate, octachlorostyrene, Kepone (with additional 12mls 1% methanol in hexane).

#### C. Silica Gel:

- 1. <u>SG Fraction I</u> (100 ml petroleum ether) n-dodecane, n-tridecane, n-tetradecane, ocylcyclohexane, n-pentadecane, nonycyclohexane, n-hexadecane, n-heptadecane, pristane, n-octadecane, phytane, n-nonadecane, n-eicosane.
- 2. SG Fraction II (100 ml 40% methylene chloride in petroleum ether followed by 50 ml methylene chloride) napthalene, fluorene, phenanthrene, anthracene, fluoranthrene, pyrene, 1,2-benzanthracene, chrysene, benzo [b] fluoranthrene, benzo [k] fluoranthrene, benzo [e] pyrene, benzo [a] pyrene, 1,2:5,6-dibenzanthracene, benzo

#### D. Silicic Acid:

- 1. SA Fraction I (20 ml petroleum ether)
  HCB, mirex
- 2. SA Fraction II (100ml petroleum ether)
  PCB's, p,p'-DDE (splits with SA III)
  - 3. SA Fraction III (20 ml mixed solvent: 1% acetonitrile, 80% methylene chloride, 19% hexane)
    alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordane, heptachlor epoxide, gamma-chlordane, trans-chlordane, toxaphene, o,p'-DDE, alpha-chlordane, p,p'-DDE (splits with SAII), o,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, dicofol.